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This is the second of a special series of reports which are being published in order to make available, for the benefit of the industries concerned, results of scientific and industrial value contained in the technical records of the Department of Explosives Supply of the Ministry of Munitions. The work recorded in these Reports was done at or in connexion with some of the National Factories during the war The preparation of the necessary abstracts of information was begun by the Ministry of Munitions at the close of the war, and arrangements were afterwards made by the Department of Scientific and Industria Research to complete them. The Department wish it to be clearly understood that the interesting information contained in this series o reports is the result of the labours of the Ministry of Munitions, and is being compiled by Mr. W. Macnab, C.B.E., F.I.C., an officer of that Ministry now attached to the Department. The first report in the series, " Recovery of Sulphuric and Nitric Acids from Acids used in the Manufacture of Explosives: Denitration and Absorption," has already been published for the Department by His Majesty's Stationery Office.

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INTRODUCTION.

Although the manufacture of TNT *per se* can have but a limited interest for the general public, yet the problems involved in devising methods for its preparation, together with their subsequent development, provide many parallels with other substances of a similar nature.

Thus, the working out of the best conditions for nitration, the purification of the finished product, the utilisation of waste materials, and the handling in bulk of large quantities of organic and inorganic chemicals are common to most branches of chemical industry.

One of the features of this manufacture which should prove of general interest is the type of plant adopted in dealing with the weak acids produced during the process, another being the methods devised to produce waste acids as free as possible from dissolved or suspended organic matter.

Under the heading "History of Manufacture" (Section I.) have been grouped the various processes which have been in use in Government factories during the period 1915-1918, and a brief general outline is given of the reasons for their adoption. For purposes of detailed description, however, it is thought advisable to take the case of the largest TNT plant, namely, that at H.M. Factory, Queen's Ferry, which was erected on an extensive scale and which embodies the results of a very wide engineering knowledge of chemical plant.

It is impossible in the scope of this account to make more than a passing reference to the mass of information resulting from the elaborate and detailed investigations carried out throughout the country both in the laboratory and on the semi works scale. The aim has been rather to indicate the object in view when such investigations were initiated, together with the lines on which the work was carried out, and to emphasise the conclusions arrived at, showing at the same time their practical application, if any, to the manufacturing scale.

Apart from a reference to the analyses of the mixed nitric and sulphuric acids used for nitration purposes, no mention is made of the actual mixing process or of the general acid cycle in use. It is intended that this subject shall be dealt with in a separate publication, which will include descriptions of the acid-cycles required in the production of TNT, nitro-cellulose, and nitro-glycerine; SO_3 and water balances; plant and process for mixing acids; etc.

In the present work the description of plant and process is made throughout as full as possible, but in one or two instances information is withheld for obvious reasons.

SECTION 1.

HISTORY OF MANUFACTURE.

General.—Trinitrotoluene or TNT was not a Service Explosive until the commencement of the war. It had been tried experimentally at Woolwich, but was manufactured only in small quantities chiefly as a minor by-product of the dye industry. The manufacture was carried on under conditions which gave very poor yields of trinitrotoluene and were inconvenient for the production on a large scale. It was therefore necessary to build up a manufacture practically from first principles, and to ascertain conditions which would give the highest possible yields of TNT in order that the available supplies of toluene might be utilised to the best advantage. The nitration of toluene to mono- and dinitrotoluene offers no difficulties. It is only in the conversion of dinitrotoluene to TNT that any difficulties arise in the manufacture.

As the existing manufacture of TNT was on a diminutive scale and was extremely wasteful, it was necessary to devise new conditions on the basis of laboratory research. The existing publications on the subject were very few in number, and offered practically no guidance to the manufacturer. The methods were very primitive, and certainly did not represent the best continental practice in the manufacture. Experience in the production of other nitro-compounds in this country was also of no assistance. To some extent the nitroglycerine process was taken as a precedent, and the manufacturing methods which had proved useful in this connection were tried in the preparation of TNT, but on the whole this precedent did not prove very helpful. Thus, the displacement process which has been of great value for nitroglycerine has not been nearly so successful for the manufacture of TNT, although it was subsequently adopted for a time at one or two factories, and de-acidification by means of alkali, which works quite successfully in the case of nitroglycerine, is altogether useless in the washing of TNT, as the molten TNT is strongly attacked by the alkali.

The Research Department at Woolwich undertook to work out the conditions for the preparation of TNT without using oleum, which was at that time extremely scarce. Some hundreds of nitrations established the best conditions of nitration, and the conditions thus arrived at were further tested in a small experimental plant with a capacity of about ¼ ton of TNT.

Trinitrotoluene differs from most of the other compounds which have been dealt with, in that there is only one main method for its preparation—viz., the nitration of toluene through mono- and dinitrotoluene to trinitrotoluene. Any methods other than those of direct nitration give such small yields that they do not come into question. The efficiency of the preparation is very largely influenced, however, by the conditions under which the TNT is made. When the methods described in previous publications were tried, they were found to give unsatisfactory yields, part of the toluene being oxidised by the nitric acid, forming large quantities of nitrous fumes. The conditions for avoiding this oxidation were, however, soon learnt, and it became possible to lay down a scheme of nitration which converted toluene to TNT with comparatively little waste, and with a minimum of oxidation and formation of nitrous fumes. The results of the Woolwich researches are embodied in a series of reports on the manufacture of TNT, and a third report on trinitroxylene followed at a later date. The main points in which these reports recorded an advance on previous knowledge were:—

(1) The importance of certain conditions of temperature during the nitration which had not been previously recognised.

(2) The extraction of the residual acids from the trinitration stage by means of mononitrotoluene (afterwards called "detoluation"). Originally the separation of the TNT was very unsatisfactory, and attempts were made to simplify it by extraction with a solvent. It was found that mononitrotoluene was an excellent solvent for TNT, and was at the same time itself partially nitrated by the residual nitric acid. Thus, by using the mononitrotoluene first as a solvent for extraction and then nitrating it to TNT, the mechanical handling was simplified, and the economy of nitric acid was improved.

(3) An elaboration of the counter-current system of nitration in such a way as to utilise the acids to the best advantage. A cycle of operations was devised which utilised the waste acids from the trinitration for the nitration of toluene, which takes place in comparatively weak acids, and a saving of acid was thus effected.

Researches on various other points, such as the use of catalysts, the influence of sulphur dioxide, nitrous acid, etc., on the nitration, did not lead to any improvements, and no features based on these experiments were, therefore, incorporated into the scheme recommended. The methods laid down at this time were adopted for the Oldbury factory, and, although some details have changed, the scheme of nitration remained almost the same as that first proposed, until the continuous process came into use. The details of the process have of course been the subject of a large amount of research at the TNT factories.

The general principles of nitration were set forth in April, 1915, in a memorandum by the Department of Explosives Supply, which shows the position of knowledge arrived at as a result of the researches at Woolwich. This memo. is reproduced below as a record of the state of development at that date, and as an outline of the general principles of the reactions.

"GENERAL PRINCIPLES OF REACTIONS.

Chemical reactions.

$$C_6H_5.CH_3 + HNO_3 \longrightarrow NO_2.C_6H_4.CH_3 + H_2O$$

Toluene. Nitric Acid. Mononitrotoluene (NT or MNT)

$$NO_2.C_6H_4.CH_3 + HNO_3 \longrightarrow (NO_2)_2.C_6H_3.CH_3 + H_2O$$

Mononitrotoluene Nitric Acid Dinitrotoluene (2NT or DNT)

$$(NO_2)_2.C_6H_3.CH_3 + HNO_3 \longrightarrow (NO_2)_3.C_6H_2.CH_3 + H_2O$$

Dinitrotoluene Nitric Acid Trinitrotoluene (3NT or TNT)

As water is formed it is necessary to add sulphuric acid or oleum as a dehydrating agent, and the completeness of the reaction depends upon the ultimate concentration of the acid mixture. If too much dilution has occurred, the trinitration will be incomplete.

II. **Strength of sulphuric acid.**—Oleum is theoretically better than ordinary strong sulphuric acid, but the latter can be used for the preparation, still giving a good TNT.

III. General methods.

(1) *Direct nitration of toluene to TNT in one stage.*

This is a very crude method of preparation, and its obvious disadvantage is that the whole of the water formed in the three stages of the nitration accumulates and dilutes the acids. Consequently at the finish of the nitration, when dehydration is most urgently needed, the acids are diluted with the whole of the water. This means that a large excess of sulphuric acid or oleum must be added to keep up the concentration. Apart from the extra expense of the acid, it is evident that in vessels of a given size the quantity of trinitrotoluene will be a smaller fraction of the total charge than if less acid were necessary. It also has the disadvantage that an unnecessarily large quantity of TNT remains dissolved in the acids.

(2) *Nitration in stages.*

Toluene—Mononitrotoluene—Dinitrotoluene—Trinitrotoluene (3 sta
Toluene—Mononitrotoluene— · · Trinitrotoluene } (2 stag
Toluene———— ———— —Dinitrotoluene—Trinitrotoluene }

(*a*) *Using fresh acids for each nitration.*—This is somewhat better than (1), as the final concentration is higher, but it necessarily uses a large quantity of acid, and retains nitro-compounds in solution.

(*b*) *Using waste acids from tri-stage for the lower stages.*—The lower stages do not require acids of such high concentration, and it is therefore possible to work in this way. This appears the soundest method of working, as it attains a high concentration at the finish without an undue expenditure of acid. It also has the advantage that it forms a convenient method of dealing with the dissolved nitrobodies, mononitrotoluene being a very good solvent for the higher nitrotoluenes, so that if the acids from a higher nitration are used for a mononitration, the higher nitrobodies are carried forward into the next operation, instead of separating out in a solid form or remaining dissolved in the waste acid. Similarly the mononitrotoluene in solution can, under suitable conditions, be washed out by a final toluene extraction, leaving the acids almost free from nitro-compounds.

IV. **Isolation of mono- and dinitrotoluene.**—It has in some cases been the practice to isolate mononitrotoluene and purify it by distillation, and to isolate dinitrotoluene and separate out the metanitrotoluene, etc., by pressing out the oily matter. Both of these methods are accompanied by loss of useful material, and would only be justified under present conditions if the impurities formed such a drawback as to render it difficult to obtain a trinitrotoluene of reasonable purity. This is not the case; hence it appears better to subject the intermediate nitrobodies to further nitration, without removing them from the nitration plant.

This opens up the point that, as the intermediate nitrobodies are not to be isolated, there is no necessity to halt at the exact molecular stages of mono- and dinitrotoluene. If for any other reason it is preferable to halt at a mixture of mono- and dinitrotoluenes represented by 1½ NT, there can be no objection to so doing.

V. **Ideal conditions.**—If the practical difficulties could be overcome, the best process would probably be a continuous counter-current system in which toluene passes in at one end and is nitrated gradually up to trinitrotoluene by nitration acids travelling in the opposite direction, viz.—

Toluene ⟶ TNT
Waste acid ⟵ Fresh acid

The concentration at the finish would then be at its maximum, and the waste acids would be completely depleted of nitrobodies. Manipulative difficulties have so far prevented the realisation of this, but such a process is quite conceivable.

In practice the process must be divided into a series of steps, which shall fulfil the following conditions, but the number of steps must not, however, be so great as unduly to increase the amount of handling:—

(1) To economise acids, partly on account of economy and cost of recovering spent acids, and partly because the higher the proportion of acids the smaller the charge of toluene in an apparatus of given size.

(2) To nitrate as highly as possible. The condition for this is that the water of reaction in the final phase shall be fairly low.

(3) To deplete the residual acids of nitric acid as far as possible, partly to economise acids and partly, if possible, to render denitration of the acids unnecessary.

(4) To deplete the residual acids of dissolved nitro-compounds as far as possible, partly to save the nitro-compounds, and partly because the latter are troublesome during the concentration process.

VI. **Choice of steps.**—The halting places in the nitration may be made in several places, the exact position of which probably does not matter a great deal so long as the main principles are fulfilled. The following considerations may be suggested as a guide to the choice of the stages of nitration.

(1) The final stages should be made as short as possible so that the water of reaction may be reduced to a minimum, leaving the final concentration high, viz., the toluene should already be fairly highly nitrated before entering on the final nitration. At the same time, it is necessary to obtain as complete separation as possible of the acids from the nitro-compounds; this entails cooling to a moderately low temperature (say,

30°-35° C.); and as it is more convenient to work with liquids rather than with solids, it is desirable that the nitrotoluene be not so highly nitrated as to solidify on cooling to this extent. This points to a mixture corresponding to about 1½ NT to 1¾ NT.

(2) At the other end of the series the waste acids are treated with fresh toluene. Conditions for this must be rather carefully regulated, otherwise a red-brown colloidal emulsion is formed; this can be avoided by dilution or by the presence of a certain excess of nitric acid.

The objects to be fulfilled in this stage are to exhaust the acids of their nitric acid as completely as possible, and to extract all nitro compounds.

(*a*) *Extraction of nitro-compounds.*

If the toluene were nitrated up to a stage approaching mononitrotoluene, the acids would remain saturated with this body, but if toluene is left in excess it will extract most of the nitrobody. From this point of view the nitration in this stage should be carried only a short way—say, up to ½ NT.

(*b*) *Depletion of nitric acid from residual acids.*

Nitric acid is necessarily present since the preceding nitration requires a certain excess. The balance of nitration on the one hand, and the extraction of organic matter on the other hand, must be regulated by the dilution. The greater the dilution, the better the extraction of nitro-compounds, but the worse the nitration proceeds. On the whole, the acids should be diluted about as little as is compatible with avoiding the formation of the colloidal suspension mentioned above.

This suggests the phases :—

$$\text{Toluene} \longrightarrow \tfrac{1}{2}\,\text{NT} \dashrightarrow 1\tfrac{3}{4}\,\text{NT} \longrightarrow 3\,\text{NT}$$

The question remains, whether the number of steps should be increased by sub dividing any of these. The first stage is hardly capable of subdivision; the final stage could only be sub-divided by departing from conditions suggested above (VI. (1)). As regards the middle phase, if it will proceed without sub-division by the use of waste acids from the final phase, there can be no object in sub-dividing it into more than one stage. It is hardly correct to speak of the above as a three-stage process, as the first stage is hardly a nitration, but only a toluene wash to get out the remains of the nitrobodies. It is essentially a *two-stage process*, in which the nitration is fairly equally divided between the two stages.

VII. **Dilution with water before separating TNT.**—It is convenient to separate the TNT from the acids in the molten condition at a temperature above 80° C. The solubility of TNT in the strong waste acids at this temperature is very high (30 or 40 per cent. of the TNT may easily remain dissolved). Hence, if the acids are undiluted, too much TNT remains in solution. It is true that this would be dissolved and carried forward by the lower nitrotoluenes, but it is nevertheless objectionable

(1) because it occupies space in the nitrator to no purpose;

(2) because it retards the nitration by diluting the dinitrotoluene and decreasing its active mass;

(3) because it is exposed all the time to oxidation by the nitric acid.

A fairly small dilution with water throws out most of the dissolved TNT and still leaves the acids strong enough to fulfil their function of effecting the lower nitration. Hence it appears advisable, before separating the TNT, to bring the total water content to something like 15 per cent.

VIII. **Addition of nitric acid.**—If the residual acid from the final stage is to effect the lower nitration, it must have sufficient nitric acid present for this purpose. This may be added in three different ways:—

(1) Addition of sufficient nitric acid to the original nitration mixture to carry through the subsequent nitrations. This might be advisable if using 100 per cent. nitric acid, as an increase in the quantity of this would improve the final nitration. But if only 92 per cent. nitric acid is available, the use of a large excess of nitric acid at the same time introduces some water, and there are limits to the amount that can be added with advantage. Hence, in this case (and particularly if oleum is not used) it is better not to add the whole of the requisite nitric acid for the introduction of three nitro-groups at the start, otherwise the TNT will probably be inferior in quality, and more oxidation will occur.

(2) Addition of nitric acid after separating the TNT.

(3) Addition of dilute nitric acid instead of water as a diluent before separating the TNT. This has the advantage of using the cheaper dilute nitric acid, and appears on the whole the most rational, though method (2) may be reverted to if (3) is objectionable on account of fumes during the separation.

IX. **Temperatures of nitration.**

(1) *Stage: Toluene* $\rightarrow \frac{1}{2}$ NT. This, being essentially an extraction, will be done at a low temperature, as near to ordinary temperature as possible.

(2) *Stage:* $\frac{1}{2}$ *NT* $\rightarrow 1\frac{3}{4}$ *NT*. This may be carried out at a temperature of about 50° C. A temperature somewhat above the ordinary assists the nitration, but too high a temperature would readily lead to oxidation.

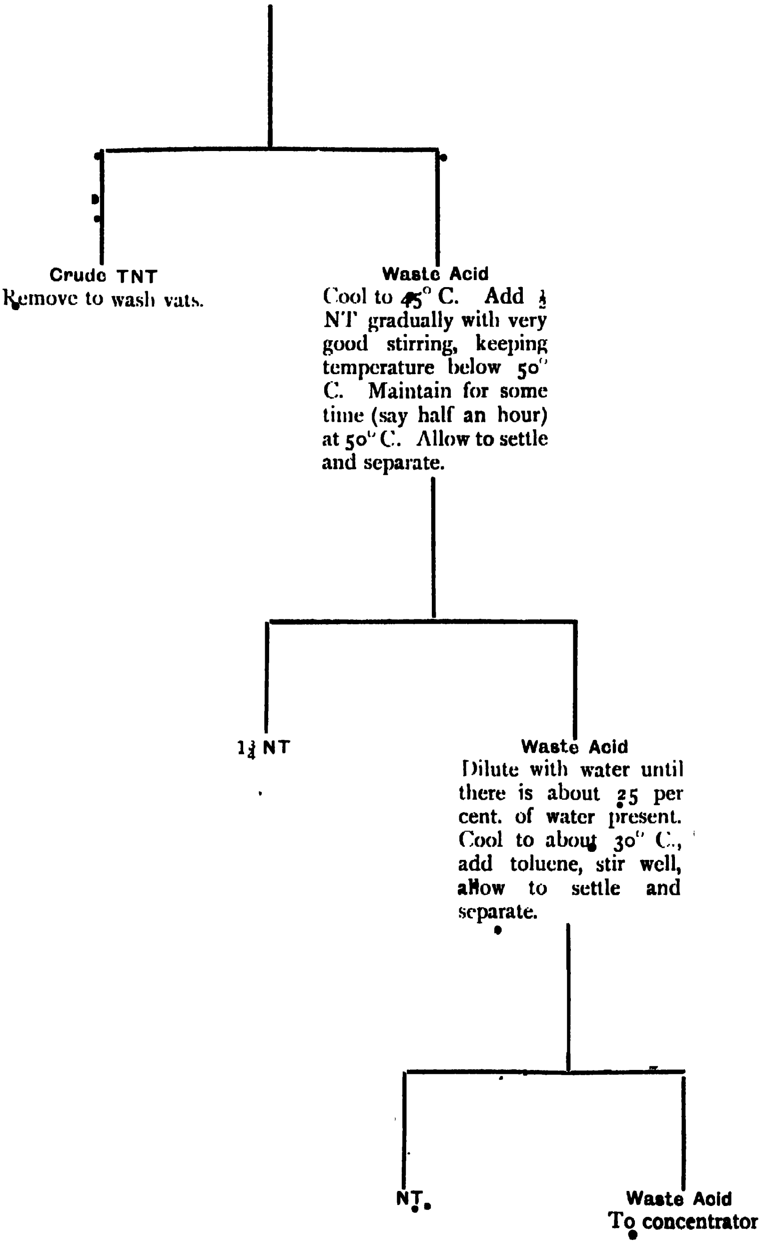
(3) *Stage* $1\frac{3}{4}$ *NT* $\rightarrow$ 3 *NT*. It is important in this stage to ensure that as little oxidation as possible takes place. The temperature coefficient of the oxidation is much higher than that of the nitration, and consequently the oxidation should be restrained by keeping down the temperature for some hours at the start. A suitable combination of temperature is the following:—

3 hours at 70° C.; 1 hour rising to 110° C.; 2 hours at 110° C.

X. **Scheme of nitration.**—The above considerations lead to the following scheme :—

1¾ NT

Add sulphuric acid (100 per cent. or 96 per cent.) to dissolve. Add mixed acids containing about 2 molecules of nitric acid. Keep the temperature below 70° C. during addition. Then maintain for 3 hours at 70° C., heat during 1 hour to 110° C., and maintain for 2 hours at 110° C. Cool to 85° C., at the same time adding dilute nitric acid (of say, 1.4 sp. gr.). Separate.



Crude TNT
Remove to wash vats.

Waste Acid
Cool to 45° C. Add ½ NT gradually with very good stirring, keeping temperature below 50° C. Maintain for some time (say half an hour) at 50° C. Allow to settle and separate.

1¾ NT

Waste Acid
Dilute with water until there is about 25 per cent. of water present. Cool to about 30° C., add toluene, stir well, allow to settle and separate.

NT.

Waste Acid
To concentrator

Phase 1 (Toluene → ½ NT) must be effected in lead vessels, as iron will not stand the dilution very satisfactorily (this may possibly not be absolutely binding).

Phase 2 (½ NT → 1¾ NT) is optional, but lead vessels would have the advantage that the dilution could be carried out before separating, and the solubility of the nitro-compounds thus decreased.

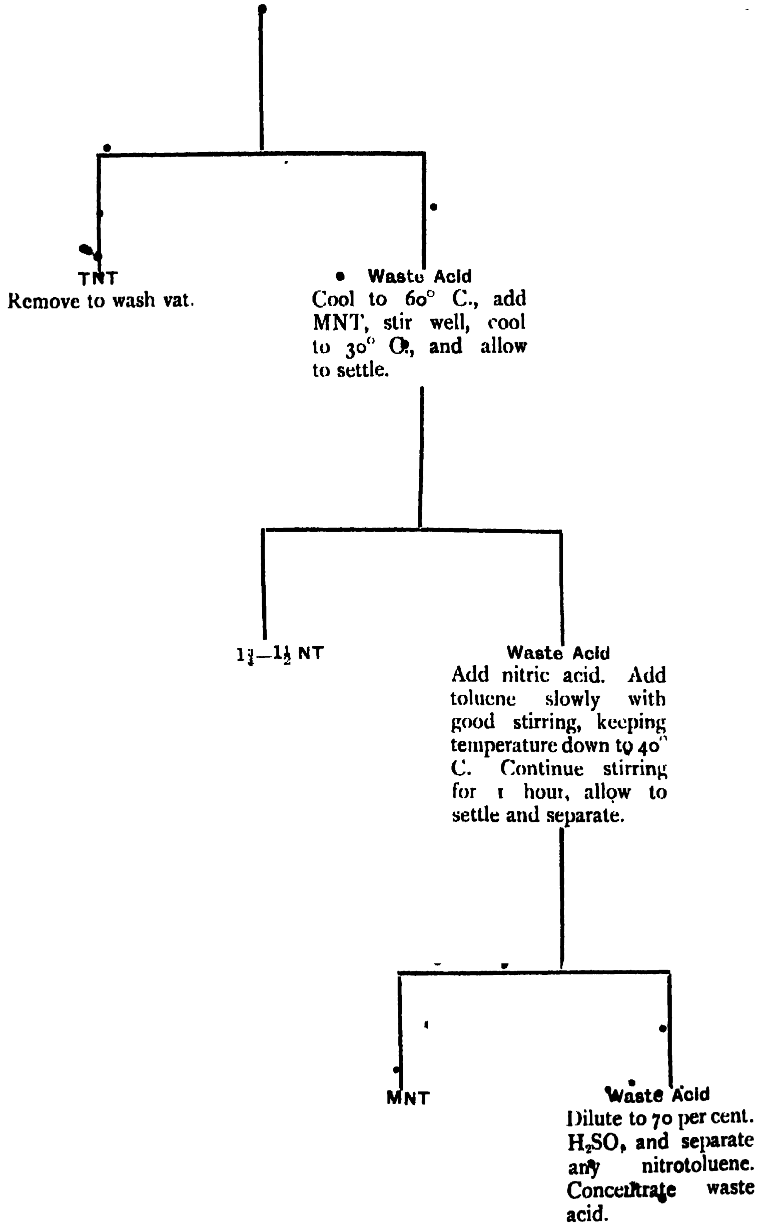
Phase 3 (1¾ NT → 3 NT) requires steam-coils under a certain pressure, and iron appears somewhat preferable, though lead could possibly be used up to the pressure required.

If no lead vessel is available, phase 1 is excluded, and it further becomes necessary to modify phase 2, because it would otherwise be impossible to cool sufficiently to separate the nitro-compounds completely. Hence the following scheme appears more suitable :—

$1\frac{3}{4}$–$1\frac{1}{2}$ NT

(Containing dissolved TNT)

Run slowly into mixed acids with good stirring, keeping the temperature below 70° C. When all is added, maintain for 3 hours at 70° C., heat during 1 hour to 110° C., and maintain for 2 hours at 110° C. Cool to 85° C., at the same time adding dilute nitric acid. Separate.



TNT
Remove to wash vat.

Waste Acid
Cool to 60° C., add MNT, stir well, cool to 30° C., and allow to settle.

$1\frac{3}{4}$—$1\frac{1}{2}$ NT

Waste Acid
Add nitric acid. Add toluene slowly with good stirring, keeping temperature down to 40° C. Continue stirring for 1 hour, allow to settle and separate.

MNT

Waste Acid
Dilute to 70 per cent. H_2SO_4 and separate any nitrotoluene. Concentrate waste acid.

April 1916."

TNT plants.—The main Government plants for the manufacture of TNT were those at Oldbury and Queen's Ferry, which were erected on an extensive scale under the direction of Mr. K. B. Quinan, and which embody the results of a very wide engineering knowledge of chemical plant. These factories have worked very successfully and produced a very large proportion of the whole of the TNT manufactured in this country.

Nitration processes in use at H.M. factories:

(1) *R.D. Process No.* 1.—This starts with MNT, which is nitrated under specified conditions to TNT. The waste acids from the TNT are detoluated* with MNT to extract the dissolved TNT, and to use up any residual nitric acid by nitrating some of the MNT to DNT.

The mononitrotoluene used in the original experiments was the product of a separate reaction, so that this process became the basis of the processes used at Oldbury and Queen's Ferry, in which the mononitration plant is quite distinct from the trinitration plant by reason of the need to nitrate toluene petrol (ex Borneo spirit), the petrol being removed from the MNT by distillation.

In the second Woolwich report, considerable developments were indicated in the use of waste acids from the trinitration process for the nitration of toluene to mononitrotoluene, after the acid was suitably fortified by the addition of nitric acid.

(2) *R.D. Process No.* 2.—In the development of processes for the manufacture of TNT without the use of oleum, it is evident that the best results in the final nitration are obtained when the nitro-compound has already been nitrated as far as it will readily nitrate in weaker acids, as the strong acids in the trinitration are then the less diluted during the course of the reaction.

A three-stage process was therefore developed as more nearly approaching a complete counter-current system. The toluene is nitrated successively to compositions roughly represented by ½ molecule NT, 1¾ molecule NT, and TNT, the waste acids stepping down to each successive stage from the trinitration with the addition of the necessary nitric acid. At the same time the waste acids from one stage are automatically washed free from the residual nitro-compound produced at that stage by the nitro-compound formed in the subsequent stage. In this way the final waste acid should contain the minimum quantity of the highest and least soluble oil, and also the minimum of nitric acid.

It is necessary for mechanical reasons to avoid the solidification of the nitration product, especially in the middle stage, at moderate temperature, and thus the degree of nitration short of the limit represented by DNT is purposely kept below the dinitro-stage.

Considerable trouble has also been experienced in the final stage, the washing of the acid with toluene and its first nitration, owing to the danger of forming a loose compound between toluene and nitrous acid (always present) which is extremely soluble, thus causing the waste acid to run away heavily charged with nitro-body. This nitro-body causes considerable trouble at the denitration plants by the formation of tarry products, and the capacity of the denitration plants is seriously reduced. This can, however, be avoided by careful attention to detail, especially to the dilution and nitric acid content of the acids at various stages.

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* See page 2.

(3) *Two-stage Process proceeding from toluene to TNT.*—In this process the toluene is nitrated successively to approximately 1¾ NT and then to TNT, the acid from the higher stage being subsequently fortified with nitric acid and used for the lower stage.

This process has been very successful from the points of view of yield, quality of product, and convenience in working. It is necessary to adjust carefully the composition of the nitrating acids, especially at the lower stage.

(4) *Continuous Process.**—This was started at Oldbury in October, 1917, as a result of exhaustive research in the laboratory and on the semi-works scale. It is a counter-current process. Strong sulphuric acid and MNT are fed in at opposite ends of the plant and emerge as TNT and spent acid respectively. The nitric acid is fed in at intermediate stages of the plant.

The plant consists of a number of pairs of cast iron pots. One of each pair is agitated and the other acts as a settling vessel. From the settling vessel, or separator, the nitro-body goes forward to the next agitator, and the acids travel in the reverse direction to the next agitator. Thus the nitro-body is continually meeting stronger and stronger acid. The vessels are fitted with coils for temperature control, and the temperature varies in the different pans, having a maximum of 100° C.

The plant has proved to have a remarkably high capacity. Although built nominally for an output of 100 tons per week, it has proved capable of manufacturing some 500 tons per week of TNT. The consumption of toluene does not differ much from that of other processes, and the advantage lies mainly in the high capacity and consequent saving of labour and capital cost of plant.

(5) *Other Processes.*—Apart from the above processes, some factories have worked on the preparation of TNT by the successive nitration of toluene to MNT, DNT, and TNT, which were separated at these stages. The successive nitrations were done either with fresh acids or with fortified spent acids.

The only other methods which have been considered are those in which nitrates were used instead of nitric acid. Sodium nitrate was tried at Woolwich and elsewhere. The main difficulty in this is the recovery of the waste acid owing to the sodium salts present. It is necessary to concentrate to a certain degree until the sodium bisulphate reaches its minimum solubility, and then to cool and filter.

A method in which ammonium nitrate was used was proposed, but this proved to have disadvantages which outweighed its advantages.

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* English Patent No. 124461.

Efficiency.—The various methods do not differ very greatly as regards the yield of TNT from toluene or in the consumption of nitric acid. The yield of TNT per part of toluene has varied from 1.8 to 2.1 parts. During 1918 it was more the exception than the rule to find a factory producing less than 2.0 parts of TNT per part of toluene. Similarly, great strides have been made in the reduction of the consumption of nitric acid, a reasonable working figure being now 1.04 parts nitric acid per part of TNT. In this respect Queen's Ferry has barely exceeded 1.00 for many months.

Very little difference in the efficiency has been observed in the nitration of coal tar toluene on the one hand and toluene from Borneo petrol on the other. Toluene-petrols from Trinidad and Persia have also been nitrated experimentally on the works scale with similar results.

Washing of TNT.

General.—The main impurities in TNT apart from mineral acids are dinitrotoluene, unsymmetrical trinitrotoluenes, tetranitromethane, nitrobenzoic acids and nitrocresols. The two last mentioned are of an acid nature and are removed in the washing process, whereas impurities such as unsymmetrical TNT require more elaborate methods for their removal (see Purification, page 18). The adhering mineral acids are easily removed by washing with water, but the nitrobenzoic acids and nitrocresols are more difficult to remove, as they are dissolved in the TNT and are not very readily extracted by water.

Originally, the washing of nitroglycerine was taken as a precedent, and attempts were made to wash out the acids by treatment with soda, but this proved to be a hopeless method. TNT is converted by alkali into an isomeric compound which forms a sodium salt, and then undergoes a radical decomposition, which is not only wasteful but dangerous, as the sodium salts of the decomposition products appear to be particularly sensitive. This decomposition takes place even when small quantities of sodium carbonate are allowed to be in contact with TNT. The washing had therefore to be effected by hot water, successive quantities of which were agitated with the TNT in lead or wooden vessels. In this way the mineral acidity was soon removed (down to about 0.1 per cent., as H_2SO_4), but to remove the organic acidity (from 0.1 per cent. to below specification 0.03 per cent.) proved to be a more difficult operation. Large quantities of water, often as much as 5,000 gallons per ton TNT, had to be used in as many as 12 or more applications, according to the efficiency with which the separation between the TNT and the water layers was made at the end of each wash. Also, the large quantities of hot water carried away and deposited considerable quantities of TNT, which involved a great deal of handling.

Much effort was spent in ascertaining such conditions of washing as would neutralise the nitrobenzoic acids, etc., without attacking the weakly acid isomer of TNT, and the two main principles resulting from a general review of the subject, and which formed an advance on previous methods were :—

1. Pelleting of the TNT in such a manner as to produce a finely divided solid which could be washed very free from acid.

2. The use of a weakly basic hydrolysed salt such as sodium acetate as an aid to the hot washing. In this way a means was found to neutralise the stronger organic acids without the addition of any alkali which would attack the TNT.*

These principles were subsequently widely adopted. The method of pelleting by dropping the hot liquid TNT into air-agitated cold water soon gave place to the method of breaking up the TNT stream by a jet of cold water. This method was used for a long time, and the bulk of the mineral acid was removed at the first pelleting operation with great ease and without waste of TNT in the wash water. It gradually became to be recognised, however, that by far the greater part of the acidity removal, after the TNT had passed the first pelleting (acidity 0.12 per cent., as H_2SO_4) was effected by the hot water treatment necessary to re-melt the TNT between the pelletings. This condition was accounted for by the fact that the pelleting removes the bulk of the mineral acidity quite readily, whereas the organic acidity is more persistent and is not amenable to a surface treatment. The pelleting processes, therefore, drifted back to the method of hot washing, with the addition of a first cold pelleting of the very acid TNT which removed the bulk of the mineral acidity in a clean light-coloured effluent, and which also saved considerable wear on vessels used in the subsequent hot washing.

Of the weakly basic salts tried, the most promising appeared to be sodium acetate, and this was used for a long time at one of the smaller factories, and subsequently at Queen's Ferry. Sodium formate was tried at Oldbury and gave equally good results. On the whole, considerable saving was made in steam and plant capacity (due to reduction in the time cycle required) through the adoption of this system, but the conditions under which it could be used were by no means standard. Exactly as with the hot washing methods, there were periods during which the acidity was peculiarly persistent, and this was at last connected with the presence of tetranitromethane, produced during the trinitration process, which varied in amount in an unaccountable manner from time to time. The only ready means of removing tetranitromethane from the TNT was by steam distillation, and this formed a strong reason for the use of the hot washing process.

Continuous Washing Process.†—This was developed at H.M. Factory, Oldbury, and formed a great advance on the previous methods as regards capacity of plant and efficiency. The plant consists of a series of pairs of pots arranged on the same principle as in the continuous nitration plant (as a matter of fact the continuous washing process was the precursor of the continuous nitration process). Molten acid TNT passed in at one end and clean water at the other. These met one another in counter-current and the outgoing products were neutral TNT and strongly acid water.

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* The dinitrobenzoic acids have affinity constants ranging from 0.002 to 0.008. Trinitro cresol has an affinity constant of about 0.16, and dinitrocresol about 0.00008, whilst acetic acid has a constant of 0.000018.

† English Patent No. 125140.

The pots were steam jacketed and provided with stirring gear. The first vessels of the series were of silicon-iron, and the later ones of lead. The lead pots were, however, strongly attacked, and were later replaced by cast iron.

The continuous process has been very successful, the water consumption being reduced to 600 gallons per ton of TNT. A similar plant was subsequently erected at Queen's Ferry, a full description of which is given in pages 80-85.

An electrolytic process of de-acidification was brought forward in which, by bringing the TNT into contact with hot water and putting a cathode in the TNT and an anode in the water with a potential difference of about 5 volts, it was suggested that the acids could be removed very completely without any agitation.

The main object of the washing is to remove acid from the TNT. It was thought, however, that by the addition of certain salts to the wash water organic impurities might be removed by a specific action of the reagents added on the organic impurities. This research did not lead immediately to the desired results, but more recently reagents having the desired properties have been discovered, viz., sodium sulphite and sodium hypochlorite. (See Purification, pages 23-26.)

Filtering of TNT.—TNT is liable to contain suspended matter. This may consist of sulphates of iron and lead, and of the so-called "transformation product" of TNT. The latter is formed from TNT in contact with basic substances and forms the "insoluble in benzene" referred to in the specification (page 18). If these are not removed, they either remain in the TNT and form a source of instability or are deposited in the various containing vessels, particularly the drying tanks through which the TNT passes in the process. If such deposits are allowed to accumulate they become extremely hard, and their removal presents a difficult problem. A fire at one works broke out in the bottom of a vessel in which such a deposit had been allowed to accumulate, and was suppressed only with difficulty.

It seems to be desirable, therefore, to filter the molten TNT as it runs away from the washing plant. The nature of the impurities arrested by the filter are such that very frequent changing is required, or the cloth becomes impervious to TNT; and the equipment must be designed to suit this condition. Steel woven cloth (100 mesh) has been found to be suitable for the filtering cradle. (See pages 100-102 for detailed description of Queen's Ferry practice.)

Drying of TNT.—Water is nearly insoluble in TNT, but not entirely so, and hence if the molten TNT be allowed to solidify without drying, it contains small quantities of water. Drying takes place easily, and the general method has been to keep the TNT molten in a large tank for some time in order to drive off the moisture. The temperature necessary for drying formed the subject of a good deal of investigation, and it was decided to fix a maximum limit of 120° C. Air may be passed through the TNT to assist the removal of moisture. It was originally feared, on the basis of statements which had been published, that the air would cause some oxidation of the TNT, but this has not proved to be a difficulty. On the whole, it was found that air blowing was liable to be troublesome mechanically, was expensive, and of little value.

Experiments were made to effect the drying by a continuous method in which TNT was allowed to flow down a coil of pipe in a counter-current of hot air. This was not sufficiently effective, but the principle was further developed at a private factory,* in which the TNT was allowed to flow over a heated flat surface, and this has proved to be an effective method of drying. Such a system has now been widely adopted, particularly as it decreases the amount of hot TNT in the driers. (See page 102.)

It has been repeatedly noticed that the volatile matter in solid TNT leaving the granulator drum is little more than half that of the liquid TNT running on to the drum. A remarkable amount of drying of solid flaked TNT also takes place during storage and transport.

Solidification of TNT.—The original method was to cast the TNT into cakes, and this continued to be used at many factories, as it provided the TNT in a suitable condition for purification with alcohol. A much more expeditious method of solidification is to take up the TNT on a rotating drum, cooled by water and provided with a scraper to remove the solidified layer of TNT. The majority of the TNT was treated in this way; by regulating the speed of the drum and the temperature of the liquid TNT, flakes of various thickness could be produced, though except for the highest grade product there was no specification limit for thickness.

The method of cooling TNT in a circular trough provided with rotating ploughs to break up the TNT as it solidified was used in America, but did not come into general use in this country.

Sources of danger in the manufacture.

(1) *Poisoning.*—The risk of poisoning among TNT workers has been found to be somewhat greater than was originally anticipated, and at one time the problem was of considerable magnitude, though suitable precautions have since avoided much of the trouble. Many girl nitration operatives have worked for two years without trouble.

(2) *Fire risks, etc.*—During mononitration there is some risk of the action becoming too violent if the conditions of mixing the toluene and the acids are not closely adhered to, and accidents have occurred from this cause. The trinitration, on the other hand, proves to be a safer reaction than was at first anticipated, if ordinary precautions are taken. According to the conditions originally described in the technical literature, oxidations can easily occur with sudden rise of temperature and evolution of large quantities of fumes, but by adhering to the scheme of temperature regulation alluded to above, very little oxidation occurs. Much discussion has taken place as to whether coils, jackets, or other means of cooling should be used, but, on the whole, there is not much difficulty in controlling the temperature. A much more serious matter is the danger of the stopping or slowing down of the stirring gear during the mixing of the charges or whilst the reaction is still very energetic.

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* Clayton Aniline Company (Brit. Pat. 9722, 1917).

Inflammations may occur through the accidental spilling of nitric acid or mixed acid on to woodwork or other inflammable material in the plant, and several fires have resulted from this cause. A fire thus started may lead to the inflammation of toluene or nitrotoluene in the nitrating vessels. Minor accidents have occurred through TNT remaining in contact with heated steam pipes, and in the distillation of benzene from TNT in the laboratory during the testing of the substances insoluble in benzene.

The greatest danger of fire is incurred if the crystallisation of TNT from inflammable solvents is carried out near to the building in which the nitration is carried on.

In consequence of several serious accidents resulting from this cause it was decided at an early date that the crystallisation or purification of TNT by inflammable solvents should not be carried out at the individual factories, but should be centralised at several works built specially for the purpose.

TNT was exempted from the Explosives Act on the 11th June, 1910, but this exemption was revoked by an Order in Council dated 22nd August, 1917.

(3) *Sparking.*—On several occasions electric potential has been discovered between various parts of the TNT plants.

Thus, at one factory sparks were observed to pass between the lid of the nitrator and the handle of the sampling bowl, which was of iron, lead-covered, and tended to scrape on the iron lid whilst being withdrawn from the pan. This phenomenon was made the subject of a thorough investigation, but it was not found possible to reproduce the above effect. It was, however, recommended that the sampling bowl should be connected by a flexible conductor to the nitrating vessel.

Shortly afterwards it was discovered at Queen's Ferry that when the contents of a nitrator were between 90° C. and 120° C. there was sufficient potential difference between a lead coil isolated in the nitrator and an earth connection to cause a feeble glow in a 4-volt lamp. This phenomenon could be reproduced at will, and was thoroughly investigated. Following on this, visits were made to a number of TNT factories to examine into the possibility of electrification, and as a general result it has been made the practice to make earth connections to the coils and any other parts of the plant which may become electrified.

(4) *Action of metals.*—The dangers arising from contact between TNT and metals are much less serious than those which result from the formation of picrates in the case of picric acid. At the same time it has been known for a number of years that trinitro compounds act as pseudo-acids and can form metallic salts indirectly by isomerisation into iso-nitro compounds. Reports have been received from time to time indicating that dangerous metallic compounds have been formed, and it is possible that some such compounds have been responsible for certain of the mishaps that have occurred in TNT factories.

It was reported from France that iron and TNT gave a brown insoluble substance which deflagrated on heating and inflamed in contact with nitric or mixed acid. It was insoluble in organic solvents, but soluble in ammonia, and the ammonia solution after evaporation gave precipitates with salts of the heavy metals. Lead and aluminium had very little action. A general enquiry was made into this subject, and similar indefinite compounds were obtained by contact with other metals than iron. Fortunately, however, the conditions that gave rise to such compounds do not often arise in actual practice.

(5) *Action of Alkalis.*—TNT reacts somewhat readily with alkalis even in dilute solution, especially if warm. The first action is to form the salt of an iso-nitro compound, but this readily decomposes with formation of brown amorphous products of unknown constitution. In contact with solid alkalis, inflammation may readily occur at comparatively low temperatures. A full investigation was made, and it was shown to be of great importance to avoid contact of TNT with any strong alkalis. Weak solutions of hydrolysed salts can be used for the washing (see page 13), but where possible alkalis are to be avoided. Even the alkalinity in hard water attacks the TNT to some extent, and it was shown that the transformation product formed is a direct function of the hardness of the water used for washing. Thus, any cleansing materials of a strongly alkaline nature should be avoided in the cleaning of vessels into which TNT is to be introduced.

Specification.—The main point of importance has been the *setting point.* Originally the melting point was determined by the capillary tube method, and a limit of 80.5° C. was in force for a time. There was, however, so little margin between this limit and the melting point of absolutely pure TNT that the specification limit was soon altered to 80° C. Soon after the outbreak of war, crude TNT was found to be suitable for amatol,* and a second grade was therefore introduced into the TNT specifications. The setting point was found to be much more reliable than the melting point for crude TNT, and a setting point of 74° C. was fixed for the crude material. For a time the capillary melting point method was retained for pure TNT, but in view of the undoubted advantages of the setting point method, this ultimately superseded the melting point entirely.

When cold alcohol washing came to be used extensively, it was found that about 40 per cent. of alcohol could be saved by purifying up to a limit of 79.5° C. instead of 80° C. This limit was introduced as an intermediate grade after firing trials had shown that it did not differ appreciably from 80° C.

Meanwhile, the nitration methods had been improved and the setting point of the crude TNT gradually rose. At about the end of 1916 it was found advisable to raise the limit for crude TNT from 74° to 76° C. on account of exudation troubles. This was met without difficulty, as the crude product was already averaging about 76.5° C. Certain of the smaller makers produced TNT below 76°, but as this could be used for purification no waste was involved.

Thus, three grades of TNT came into use, the specification limits and actual average setting points being as follows:—

	Grade I.	*Grade II.*	*Grade III.*
	°C.	°C.	°C.
Actual average	80.2 ...	79.8 ...	76.7
Specification limit	80.0 ...	79.5 ...	76.0

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* Mixtures of TNT with ammonium nitrate in varying proportions.

The *volatile matter* is specified as 0.25 per cent., and this figure has, with a few exceptions, been met without difficulty. Latterly, the factories were instructed to reduce the volatile matter to 0.1 per cent., although this did not enter into the specification, as moisture had been found to cause clogging in melt fillings of amatol.

The *acidity* has frequently been a matter of some difficulty, as very thorough washing is necessary to reduce the acidity to the specification figure of 0.03 per cent., calculated as H_2SO_4.

The *organic matter* insoluble in benzene was found to be of importance, as it affected the stability of the TNT. Difficulty has been experienced at times in meeting the limit of 0.1 per cent. (see action of alkali, page 17).

The *ash* does not present a serious problem, as it settles out very completely from the molten TNT during the drying stage. All grades of TNT must be free from grit and must give a clear melt.

Purification of TNT.—The impurities in TNT (apart from moisture, and traces of mineral acids which are easily removed in the washing process) are:—

Dinitrotoluene;
Unsymmetrical trinitrotoluenes;
Nitrobenzoic acids;
Nitrocresols;
Tetranitromethane.

Dinitrotoluene results from incomplete nitration, and is practically always present in small quantity (mainly 2 : 4-DNT and a little 2 : 6-DNT).

The unsymmetrical isomers result from a small proportion of metanitrotoluene which is formed in the mono nitro-stage. Trinitrotoluene can exist in six isomeric forms, all of which are known :—

(1)	2 :4 :6-trinitrotoluene,	m.p.	81.0°C.
(2)	2 :3 :4 ,,	,,	112.0°C.
(3)	2 :4 :5 ,,	,,	104.0°C.
(4)	3 :4 :5 ,,	,,	137.5°C.
(5)	2 :3 :5 ,,	,,	97.2°C.
(6)	2 :3 :6 ,,	,,	79.5°C.

Of these, No. 1 is the ordinary symmetrical or TNT, and Nos. 2 and 3 result from the nitration of metanitrotoluene.

Ternary melting-point diagrams for the α, β, and γ trinitrotoluenes were drawn up by the Research Department, Woolwich, and proved of great use when applied to the study of purification problems.

An attempt was made at Oldbury and elsewhere to separate the metanitrotoluene from the mixture of mononitrotoluenes by various methods. The best results were obtained by a combination of fractional distillation and freezing out. The war was too far advanced to permit of this being put into operation on the large scale, but as a method it possessed many good points, and would probably have been very economical.

The separation of paranitrotoluene and its further nitration to pure TNT was also proposed.

The centrifuging of dinitrotoluene to remove the meta derivatives has also been under consideration, but has the disadvantage that the valuable 2 : 6-isomer is lost.

The nitrobenzoic acids and nitrocresols are removed in the water washing.

Tetranitromethane should be removed in the water boiling treatment, but is generally still found in the finished crude TNT.

Objects of Purification.

These fall under three headings :—

(1) To secure better detonation.
(2) To secure greater stability in amatol.
(3) To avoid exudation.

(1) *Detonation.*—When TNT is mixed with ammonium nitrate the detonation of the mixture is equally good with crude or pure TNT, but when the latter alone is used the detonation is distinctly better when the TNT is purified. For this reason pure TNT is used in shells containing TNT unmixed with ammonium nitrate, and especially in exploders.

(2) *Stability.*—Difficulties were met with at times owing to interaction between impure TNT and impure ammonium nitrate, being due to the action of the unsymmetrical isomers of TNT. Thus, pyridine and thiocyanate react at comparatively low temperatures, but the saline impurities do not react unless the temperature is above the eutectic point (110° C. for NH_4NO_3, NH_4Cl, and $NaNO_3$). Thus the difficulty does not arise if impure ammonium nitrate is used with pure TNT, or if impure TNT is used with pure ammonium nitrate. In the case of amatol 80/20* the stability hardly comes into question, but in 50/50 and 40/60 where high temperatures are involved, it is necessary that either the TNT or the ammonium nitrate be pure.

(3) *Exudation.*—Crude TNT contains a certain percentage of impurities, consisting chiefly of dinitrotoluene and unsymmetrical isomers of trinitrotoluene. These form a eutectic of low melting point, and, hence, if the TNT is exposed to hot climates, some exudation occurs. This fact led to some difficulty during the summer of 1916, and in consequence of this there was a demand for a largely increased quantity of purified TNT for use in amatol. The difficulties due to exudation are met with under the following combination of conditions :—

(*a*) Low melting point of TNT.
(*b*) Only in amatol 40/60; amatol 80/20 does not give rise to this difficulty.
(*c*) High temperature of storage.

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* In referring to the composition of amatol the first figure always gives the percentage of ammonium nitrate present, and the second figure gives the percentage of TNT. Thus, 80/20 denotes 80 parts NH_4NO_3 and 20 parts TNT.

(*d*) Presence of exploder or smoke composition consisting of substances which are affected by the exudation.
(*e*) Absence of containers to exclude exudation.

The effect of the exudation on TNT exploders is to impregnate the TNT with the exuded matter and so deaden its explosive properties.

Methods of Purification.

General researches on the methods suitable for purifying TNT were carried out both at Woolwich and at the factories. The methods include :—

(1) Purification by organic solvents.
(2) Purifications by sulphuric and nitric acids.
(3) Purification by hot water.
(4) Centrifuge methods.
(5) Pressure methods.
(6) Purification by aqueous solutions.

(1) *Purification by Organic Solvents.*—The original method was to crystallise the TNT from an organic solvent, and this method gives a practically pure product. A mixture of alcohol and benzene was generally used, for neither of them used alone is really satisfactory, the one by reason of its poor solvent power for TNT, the other because of its excessive solvent action. The TNT was dissolved in the hot solvent and cooled with stirring, the nature of the impurity and the physical condition of the TNT being immaterial, as the TNT was completely dissolved in the hot solvent.

The main practical difficulty which presented itself in the application of this method on the large scale was the tendency of the TNT to deposit in crusts on the cooled walls of the pans and to retard further cooling owing to its extremely low conductivity. Various methods were proposed to overcome this difficulty, amongst which were the following :—

(*a*) Scrapers, chains or brushes to scrape off the crusts.
(*b*) Shallow labyrinth with shovel scrapers.
(*c*) Low temperature gradient between TNT and cooling jacket.
(*d*) Treatment of molten TNT with hot alcohol.
(*e*) Dissolve in xylene, etc., and precipitate by alcohol.
(*f*) Crystallise from small quantity of MNT and wash out with alcohol.
(*g*) Same as (*f*) except with nitrobenzene and washing with carbon tetrachloride.
(*h*) Extremely rapid stirring and "shock" cooling.
(*i*) Cooling of alcohol by evaporation in a vacuum.
(*j*) Mix hot alcoholic solution of TNT with refrigerated alcohol.

The only method known at this early period which did not involve crystallisation was the washing of ground TNT with cold alcohol, which washed away the impurities. The purity of the resulting TNT was dependent in a high degree on the state of aggregation of the material before treatment. For the best results the TNT should be slowly cooled so as to give a crystalline product, in which the isomerides and dinitrotoluene are wholly on the surface of the pure TNT crystals and are thus easily removed by cold alcohol.

At this period the purification was being carried on in a number of factories by crystallisation. The purification was undoubtedly the most dangerous part of the manufacture, but as convenient methods which did not require inflammable solvents were not known, there appeared to be no remedy for this. A series of explosions at TNT factories rendered drastic action necessary, and it was decided to stop the purification process at practically all the TNT factories, and to establish separate purifying stations to which the crude TNT from the nitration factories should be conveyed from a distance. One factory was already established on this basis, and and one for crystallisation with vacuum cooling. These factories mainpurifying plants were erected, one for purification by cold alcohol washing, and one for crystallisation with vacuum cooling. These factories maintained the supply of pure TNT until a disastrous explosion destroyed the latter plant. The other factory was operated very efficiently right up to the end, though its output became gradually less important as arrangements were made to purify TNT without the use of alcohol, thus avoiding the great danger of fire and the inevitable waste in the recovery of alcohol.

The distillation of alcohol from TNT residues was the chief danger point in the process. Nitrous fumes were frequently observed in the alcohol vapour, and for this reason a little urea was added to the alcohol before distillation. The fumes were probably due to the formation of ethyl nitrite from the TNT isomers, probably assisted by the impurities in the denaturing agent in the alcohol. The question of other denaturants in place of crude wood spirit was fully gone into with the excise authorities. The alternatives were benzene, petrol and TNT.

Solvents other than alcohol have been used to some extent, such as benzene and toluene. Others were also proposed, such as carbon tetrachloride and butyl alcohol.

(2) *Purification by the use of Acids.*

(*a*) *Sulphuric Acid.*—TNT can be crystallised from strong sulphuric acid, and this has formed the basis of a series of purification methods. The main points which require consideration are :—

(1) Economy of sulphuric acid.
(2) Accumulation of unsymmetrical trinitrotoluenes in the mother liquor, rendering it unsuitable for nitration purposes.
(3) Difficulties of filtration and the handling of strongly acid liquors.

The principles on which the following methods depend are best understood by considering that when strong sulphuric acid or the residual nitration acids are in contact with hot molten TNT the impurities are distributed between the TNT layer and the acid layer. If, however, the whole mass be cooled, the solid TNT ceases to be a solvent for the impurities, and under suitable conditions these may be caused to pass into solution in the acid. The various methods which have been suggested are as follows :—

(i) Simple crystallisation from strong sulphuric acid. This requires a large quantity of sulphuric acid, and is therefore wasteful.

(ii) Crystallisation from residual nitration acids. The impurities accumulate in the acid and so render it, in part at least, unsuitable for further nitration processes. The filtration and general manipulation of the large quantity of acid liquor make the various modifications of this process inconvenient as a technical method.

Experimental work was carried out on these processes, but none were adopted, except in a small way, where a plant lay-out was favourably disposed to the working of the process. It is not now likely that such processes would be considered for future work as compared with the newer methods using aqueous solutions.

(iii) The cold washing of TNT with sulphuric acid was put forward by Woolwich, and is analogous to the method of cold washing with alcohol. For successful treatment it is necessary to have a well crystallised TNT (crushed "toffee") through which concentrated sulphuric acid is allowed to percolate. Usually this method gave good results when the TNT had originally a very low setting point, but, on the other hand, with an original high setting point the results were not so well defined. A setting point of 79° C can be obtained from almost any quality of crude TNT by this means, but this figure represents approximately the limit of purification that can be readily obtained on a works scale. The disadvantages which present themselves are the trouble of handling material saturated with concentrated sulphuric acid, the difficulty of filtration, and the general slowness of the operations.

The process is wasteful of sulphuric acid, but the losses of TNT are not high, especially if the recovery of residues from the washing liquid proved to be feasible.

(*b*) *Nitric Acid.*—Following the successful development of the nitric acid purification method for similar compounds, it was shown that the same method could be applied to TNT manufacture, and experiments were made on this method at Queen's Ferry. In view, however, of the quantities of TNT requiring to be purified, and the difficulty of handling the large quantities of nitric acid involved, the work was not proceeded with.

(3) *Purification by Hot Water.*—It was claimed by one private firm that the impurities present in TNT can be removed by simple hot water treatment under suitable conditions. As the impurities are insoluble in water, and are scarcely attacked chemically by hot water, their removal is possibly purely mechanical.

(4) *Centrifuge Processes.*—The dinitrotoluene and TNT isomers give rise to a low melting eutectic. The removal of this by centrifuging the crude material (in the form of lumps of TNT "toffee") was proposed, and the method as it stood gave very indifferent results, but it was developed after a very full investigation, and ultimately gave extremely promising results. In its improved form it consisted in centrifuging the coarsely ground TNT and directing a jet of warm water on to it in the centrifuge. The best results were obtained when the process was done in two stages at different temperatures. The liquid from the high temperature stage was returned to the process.

An improvement on the above centrifuge process was the "phenol process." In this the TNT was melted with phenol and allowed to cool slowly. The phenol formed a eutectic with the impurities in the TNT, and this was whizzed out and the residual phenol washed out with water. By this means a setting point of 80° C. could be reached.

(5) *Pressure Methods.*—These depended also on the removal of the eutectic by physical means. The TNT was warmed gradually in a cylindrical vessel fitted with a plunger applying pressure to the TNT to express the juice. The loss of TNT was, however, somewhat high, and it was difficult on the larger scale to bring the mass of non-conducting TNT to uniform temperature, which was of course very necessary.

(6) *Purification by Aqueous Solutions.*—TNT is always washed very thoroughly with water after the nitration to remove adhering mineral acids and as much as possible of the organic acids. The other impurities, such as dinitrotoluene and the unsymmetrical trinitrotoluenes, are not readily removed by water owing to their insolubility. These compounds are not very reactive, and, although some experiments on salt solutions had been made, it was not until 1916 that suitable reagents, viz., sodium hypochlorite and sodium sulphite, were found.

(1) SODIUM HYPOCHLORITE PROCESS.—This process, which was developed and used for some time by the Clayton Aniline Co., consists of the treatment of the crude TNT with sodium hypochlorite solution (commercial "chlor liquor"). The elevation of the setting point obtained varies from 1°-1½° C.; thus the purification brought about is much more limited than that produced by the sulphite process (*vide infra*). The convenience of the method lies in the fact that the hypochlorite can be introduced during the hot water wash of the TNT, and thus no additional plant is required. The exact chemical reactions between unsymmetrical TNT and sodium hypochlorite have never been definitely worked out. Apparently other impurities also are oxidised away, and the finished TNT has a very pale colour and a very low content of organic matter insoluble in benzene.

(2) SODIUM SULPHITE PROCESS.—This process was discovered and first used in France. The method as then used (1916) consisted in casting TNT into blocks, crushing the coarse crystals formed by the slow solidification of the TNT, and washing with a solution of sodium sulphite, which is obtained as by-product from the manufacture of synthetic phenol. This process was inspected by representatives of the Explosives Department in July, 1916, and shortly afterwards experimental work on the lines indicated by the French was started at Woolwich and elsewhere. Experiments on the works scale were made, and the conditions necessary for a successful working of the process established, but the great disadvantage proved to be the plant, time and labour involved in preparing the TNT in a condition suitable for sulphite treatment, especially as the lay-out of TNT factories in this country was arranged almost entirely for flaked product, which material, having been suddenly chilled, was unsuitable for the process.

The purification depends upon the interaction of sodium sulphite with the unsymmetrical isomers of TNT in which one nitro-group is replaced by a sulphonic acid group. The sodium salts of these sulphonic acids are very soluble, forming a deep red solution, and are easily removed in the wash liquor. Symmetrical or α TNT itself is attacked to a greater or less extent according to the temperature used for the washing, though the decomposition is very slight compared with that of the unsymmetrical isomers. Sodium sulphite solution from the synthetic phenol works frequently has a strongly alkaline reaction, and in order to overcome this, experiments were made with sodium sulphite, containing sodium bisulphite, but this mixture was not so reactive as the above material. Sodium sulphite from synthetic phenol manufacture has therefore been used for the purification, and no deleterious effects have been observed. The dinitrotoluene present in the crude TNT is not attacked by sulphite; thus, the sulphite treatment does not afford a complete purification of the TNT, the measure of the purification depending upon the degree of nitration of the crude product.

In July, 1917, information was received of a modification of the original French process which was widely adopted in France and subsequently in this country. As pointed out above, the great disadvantage of the original scheme was the difficulty of bringing the TNT into a physical condition suitable for the sulphite treatment. The new process provided for a preliminary washing of crude material by ordinary hot water to remove mineral acidity, and the solidification of the fused TNT under water by a process of slow cooling with stirring. In this way there was obtained a wet magma of small but well-defined crystals of pure TNT, impurities, as in the case of "toffee," being concentrated on the surface of the crystals. When the TNT had cooled sufficiently the sodium sulphite solution was added with stirring. The reactions indicated above take place completely during half-an-hour's stirring, and on removal of the deep red wash liquor and subsequent water washing of the crystals, α TNT, together with some dinitrotoluene, remains.

The solution of sodium sulphite used contained 10 per cent. of Na_2SO_3 and was added in sufficient quantity to give 4·5 per cent. of Na_2SO_3 on the crude TNT present. It is best to add the sulphite at a temperature of 35°-40° C., but it can conveniently be added at temperatures up to 60° C., which saves time on the cooling of the charge, but somewhat increases the attack on the symmetrical TNT.

The chief mechanical difficulties have been due to the great strain on the stirrers, blocking due to too sudden cooling when cast iron vessels were used (remedied by using wooden vats), and the formation of emulsions due to colloidal iron in the TNT (remedied by adding a little sulphuric acid to the wash water).

It was also arranged to give an acid wash to the TNT (after removal of the sulphite liquor) whilst in the molten state in order to ensure complete absence of alkalies. Whilst this is the standard process, it is not at all certain that such an operation is necessary; if it could be avoided and the crystals used without remelting, a great simplification of the operations could be brought about.

The increase in setting point obtained by purification is approximately 2.3° C., and as dinitrotoluene is not affected during the process, the setting point of the final product depends upon the degree of nitration of the crude material. The loss in the purification amounts to 7-8 per cent. on the TNT used.

If any aggregates of TNT are formed during the crystallisation stage these are imperfectly attacked, as they are not penetrated by the sulphite solution. Thus the smaller particles are purer than the larger lumps. This suggested that if the sulphited product were sieved, the TNT which passed through would be of better quality than the bulk. This was tried at Queen's Ferry, and the crystals which passed through the sieve gave a setting point of 80.1° C., or 0.3° higher than the bulked batch.

A full description of the plant operations is given in pages 85-100.

Treatment of the Sulphite Mother Liquor.—Experiments have been made with the view of recovering valuable organic material from the red mother liquors from the sulphite process. Such material can be recovered, but many difficulties arise in considering large scale recovery, chiefly due to the excessive dilution of the liquor. Acidification of the liquor precipitates a small proportion of the soluble contents as symmetrical TNT, but it has no effect on the substances which arose from the TNT isomers. On treatment of the mother liquor at about 70° C. with methylamine, a mixture of dinitromethyltoluidines is obtained, which could be nitrated up to trinitrotolylmethylnitramine. The price of methylamine up to the present is prohibitive, and the sulphite liquors have so far run to waste.

Treatment of TNT Residues obtained from the Purification Process.

When TNT is purified by means of solvents or acids, the impurities are obtained in the form of a residue containing a certain quantity of TNT, together with dinitrotoluene or unsymmetrical trinitrotoluenes, or both, according to the nature of the process. The utilisation of these impurities has formed the subject of a good deal of investigation, but they are not very suitable for direct application to explosive purposes. Mixtures with ammonium nitrate, sodium nitrate, barium nitrate, ammonium perchlorate, etc., have been tried at Woolwich, but are either unstable, insufficiently powerful, or unduly sensitive. The re-nitration of the residues direct is a matter of some difficulty, owing to the isomers and the presence of small quantities of alcohol and other oxidisable impurities. Attempts have therefore been made to separate the components of the residues. Direct crystallisation methods are not satisfactory, but better success has been obtained through treatment of the residues with sodium sulphite. The most important source of such residues is from the alcohol purification process. Besides pure TNT, dinitrotoluene, and the unsymmetrical trinitrotoluenes, other impurities also are present, which appear to arise from the action of basic matters such as pyridine in the alcohol (introduced by denaturing), on the isomeric trinitrotoluenes. It was found that treatment with sodium sulphite solution at from 40°-50° C. removed the unsymmetrical trinitrotoluenes, also any impurities which are soluble in the alkaline liquors formed during the treatment; in addition, the process of washing gets rid of any traces of alcohol in the residues. In this way a crude mixture can be obtained of pure TNT and dinitrotoluene which can be nitrated direct to a good quality of crude

TNT. The sulphited residues generally still contain foreign organic bodies, which lead to a somewhat heavy formation of nitrous acid during the trinitration, and at the factory where the large-scale treatment of residues was carried out, it has been found advisable to mix this recovered product with straight dinitrotoluene to the extent of some 25-30 per cent., which makes the trinitration less troublesome. Some quantity of the 2 :3 :4-isomer has been isolated, and this substance has some value as an intermediate for the manufacture of dyes. If the residues are allowed to crystallise slowly over a matter of days, a large amount of this 2 :3 :4-TNT, together with dinitrotoluene, separates out. Subsequent treatment of this mixture with sulphuric acid removes the dinitrotoluene and some other impurities, and the 2 :3 :4-TNT is obtained reasonably pure.

SECTION 2.

PLANT AND PROCESS FOR THE MANUFACTURE OF TNT.

General.—The plant at H.M. Factory, Queen's Ferry, was designed originally for the nitration of toluene to TNT in two stages (see page 10).

The first stage consisted of a mononitration, using toluene-petrol or coal-tar toluene as the starting material. Mononitrotoluene (MNT) was prepared from toluene-petrol by nitration, followed by washing and distillation to separate the unchanged petrol. In the manufacture of MNT from coal-tar toluene, however, the operations of washing and distilling the nitrated product were unnecessary, and the latter underwent no further treatment before being used in the trinitration process. The MNT prepared from toluene-petrol was known as "pure"* MNT, and that produced from coal tar toluene as "unwashed" MNT. The MNT from either of these sources was then used for TNT manufacture, being first employed to wash the waste acid resulting from the trinitration stage. This operation is known as "detoluation." Its effect is to extract the dissolved TNT, to utilise any residual nitric acid by nitrating some of the MNT to dinitrotoluene, and to leave the acid clear and suitable for denitration and concentration. The solution of mononitrotoluene containing TNT and dinitrotoluene (under the name "acid-MNT") was then used for trinitration.

The second stage consisted of the nitration of the "acid-MNT" to TNT in one operation, using a mixed acid of low water content.

Thus, as will be seen from the above outline of the original Queen's Ferry practice, the detoluation process was introduced essentially as a washing stage, and not as a nitration stage, the only nitration effected being that resulting from the HNO_3 content (approximately 2.5 per cent.) of the waste acid from the trinitration. The acid leaving the detoluators ("spent acid") had the average composition :—

	Per cent.
H_2SO_4	72.0
HNO_3	trace
HNO_2	3.5
Soluble in ether	0.7
H_2O	23.8
	100.0
Specific gravity	1.660

The acid-MNT resulting from this detoluation process had a gravity of 1.25-1.30/25° C., and consisted mainly of MNT, with comparatively small quantities of TNT and dinitrotoluene.

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*The term "pure" refers only to the purification which the nitration product underwent during the washing and distillation stages, the final product being actually a mixture of all three isomers of mononitrotoluene.

The nitro-body content of the spent acid, given above as 0.7 per cent., was almost entirely in the form of mononitrotoluene, and no difficulty was experienced in pumping acid of this composition through blockage of delivery pipes due to solidified nitro-compounds.

As the urgent need for strict economy in the usage of nitric acid became more and more apparent, the effort to recover as completely as possible the oxides of nitrogen liberated during the nitration processes resulted in the installation of very efficient absorption systems, with the result that considerable quantities of weak nitric acid (containing 50-55 per cent HNO_3) were recovered. In addition to the above, large quantities of weak acid were available from other sources in the factory, and, as this acid could not be disposed of conveniently in any other way, it was decided to utilise it in the detoluation process, making the latter to all intents and purposes a dinitration stage. In its final form the detoluation process was so arranged that the acid-MNT resulting from it (or DNT, as it was then called), had a specific gravity of approximately 1.35, corresponding to a high content of dinitrotoluene.

The advantages resulting from the extra nitration in the detoluators are obvious, viz., economy in the use of acid, partly on account of economy and cost in recovering spent acid, and partly because the higher the proportion of acids, the smaller the charge of toluene in an apparatus of given size; and also to nitrate as highly as possible, the conditions for this being that the water of reaction in the final phase shall be fairly low.

At the same time, however, the primary object of detoluation was no longer attained, viz., the production of a spent acid of low nitro-body content, since the increased solubility in sulphuric acid of the nitro-compounds (due to the presence of more dinitrotoluene) raised this content to considerably over 1 per cent. This resulted in large quantities of valuable organic material being transferred in the spent acid to the Acids Section of the factory, thereby causing much inconvenience at the denitration and concentration plants. Frequent blockage of the spent acid pipe-lines also occurred, especially in cold weather, owing to the high percentage of dinitrotoluene in this organic matter.

In order to overcome this difficulty, additional plant was installed in order to wash the acid leaving the detoluators with MNT, and as this process actually took the place of the original detoluation scheme, it was known under the name "super-detoluation."

The above resumé summarises very briefly the development of the Queen's Ferry process of manufacture, and a fuller description is given in the detailed account which follows.

MONONITRATION.

General description of MNT section.—The MNT plant,* which is adjacent to the main TNT plant, consists essentially of 3 units. Two of these units are designed for utilising toluene-petrol, and consist of a nitrating

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* This section of the plant was designed and erected by the Asiatic Petroleum Company, which firm was responsible for production until June, 1918, when the management was handed over to the Superintendent of the main factory at Queen's Ferry.

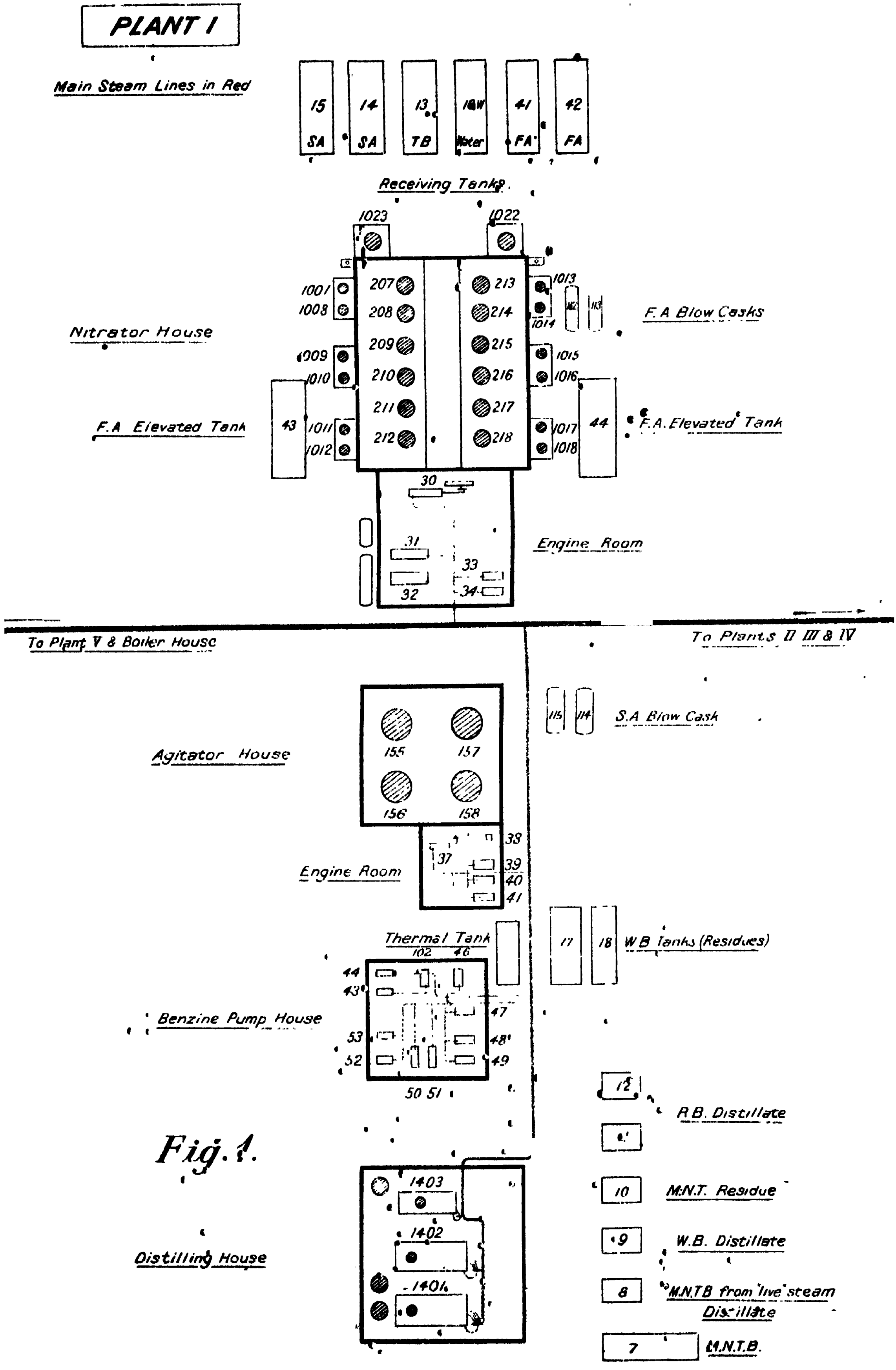
PLANT I
Main Steam Lines in Red
15 SA
14 SA
13 TB
16W Water
41 FA
42 FA
Receiving Tanks.
F.A Blow Casks
Nitrator House
F.A Elevated Tank
F.A. Elevated Tank
Engine Room
To Plant V & Boiler House
To Plants II III & IV
S.A Blow Cask
Agitator House
Engine Room
Thermal Tank
W.B Tanks (Residues)
Benzine Pump House
50 51
R.B. Distillate
M.N.T. Residue
Distilling House
W.B. Distillate
M.N.T.B from live steam Distillate
M.N.T.B.

Fig. 1.

house, a wash house, and a distillery; the third unit is especially adapted for the nitration of pure toluene, for which no distillation is necessary, so that this unit consists only of a nitration house and a separator house. In the following pages the plant and working of one unit will be described, a brief reference being made to any variations of process or plant on the other two units.

The dimensions of one MNT unit are 450 ft. x 150 ft. Fig. 1 gives a rough sketch of a typical unit and shows the relative positions of the various houses, storage tanks, etc.

About eighty yards from the actual process buildings and completely enclosed by earth-banked brick retaining walls 5 ft. high are two "tank yards," in which are situated large cylindrical steel tanks which are used for storing the various raw materials and finished products. Each tank yard is approximately 220 feet square and contains two 1,400-ton tanks, one (or two) 500-ton tanks, and three 70-ton tanks. The pipe-lines for pumping from and to the tanks pass through the surrounding walls and are concreted in. Each tank is provided with a water spray delivering on to the centre of the domed cover; each of these drenching pipes has its own control valve, operated from outside the tank yard, so that in the event of fire any desired tanks may be kept cool and isolated from the fire by a water curtain. The drainage water from these sprays leaves the tank yard by means of a brick trap, so designed that, while water can pass through, any oils, if present, are retained.

The process buildings consist of steel frames sheeted with corrugated iron, the whole being erected on a concrete foundation. The corrugated sheeting does not quite reach to the level of the concrete floor, as a low brick retaining wall is built round each house. Each house, the tank yards, and also the various pipe tracts are "welled" in this manner, so that in the event of a spill or fire the liquids are kept within the enclosure and cannot spread to unaffected buildings.

Special precautions against fire are also taken throughout the lighting system, all wires and lamps being entirely enclosed and operated by switches from the power house.

General description of the MNT process in which toluene-petrol is the starting point.—The material used is a mixture of toluene with various paraffins and napthenes, boiling round about 110° C. This mixture is known as toluene-petrol, or, in accordance with continental usage, toluol-benzine. It is obtained by distillation from "Borneo straight-run," which is the fraction coming over up to 200° C. when crude Borneo oil is distilled without the refining column. This "straight-run" is distilled in stills provided with columns of the Heckmann pattern. In the first distillation the fraction boiling from 90°-130° C. is separately collected. This fraction is then subjected to a second distillation, the fraction boiling from 95°-115° C. obtained in this second distillation being the toluene-petrol employed for the nitration. This material contains from 55 to 60 per cent. of pure toluene, and is altogether free from olefines and other compounds which would react with the mixed acid. It is conveyed to the factory either by tank car or by lighter, and on arrival is pumped into the large storage tank. During the nitration process it is nitrated by sulpho-nitric acid, which is mixed from fresh nitric and sulphuric acids and pumped to the MNT plant from the main Acids Section of the factory. The acid reacts with the toluene, but leaves the petrol practically untouched. The spent acid on standing separates out below the MNT-B* mixture and is run off and returned to the Acids Section for denitration and concentration. The acid MNT-B is washed with water until free from acid, and it is then pumped into a still, where, by means of indirect steam and finally live steam, the whole of the petrol is driven off, leaving a residue of "pure"† MNT in the still, which is pumped to storage tanks and thence as required to the TNT section (see flow-diagram, Fig. 2).

The petrol is rectified and returned to the suppliers.

The unwashed MNT process in which coal tar toluene is the starting point.—The toluene obtained in this country from coal tar, viz., from gas works and coke ovens, is almost entirely free from paraffins. It is, in fact, very rare to find as much as ½ per cent. of paraffin in coke oven toluene. Toluene from gas works tar may occasionally contain small quantities, and material obtained from tar supplied by gas works which employ vertical retorts especially may contain between 4 and 5 per cent. of paraffins. A very small proportion only of the supplies, however, is drawn from gas works operating with vertical retorts, and it is therefore possible almost completely to submerge the paraffin content of this toluene by mixing it in large storage tanks with the enormous quantities of paraffin-free toluene obtained from other sources. Occasionally batches of toluene containing 3 or 4 per cent. of paraffins have been received at the factory in cases where mixing in bulk to reduce the paraffin content has not been expedient. In these cases considerable difficulty always arises during nitration, and low yields and high acid usage generally follow. It has been found at H.M. Factory, Queen's Ferry, that paraffins present in the toluene remain in the MNT produced by mononitration, and from this material a hydrocarbon of specific gravity 0.73 and boiling at 120°-124° C. has been isolated. This material is gradually oxidised during the trinitration stage, and when it is present to any considerable extent always increases the nitric acid consumption in the manufacture and renders it difficult to reach the desired setting point.

In dealing with toluene containing quantities of paraffin less than those associated with the toluene in the toluene-petrol referred to above, it would appear necessary to fix a point beyond which increase in the paraffin content can only be dealt with on the lines described above in dealing with toluene-petrol, viz., washing the nitrated product until free from acid, and subsequent distillation to remove unchanged petrol. It appears that with a paraffin content up to 4 or 5 per cent. it would be inexpedient to wash and distil the product obtained in the mononitration stage, since the cost of this would be almost equivalent to the additional cost of nitric acid required to obtain a satisfactory product from such toluene. In the case of a toluene containing more than 5 per cent. of petrol, however, it would probably be advisable to follow the procedure used in dealing with toluene-petrol, viz.,

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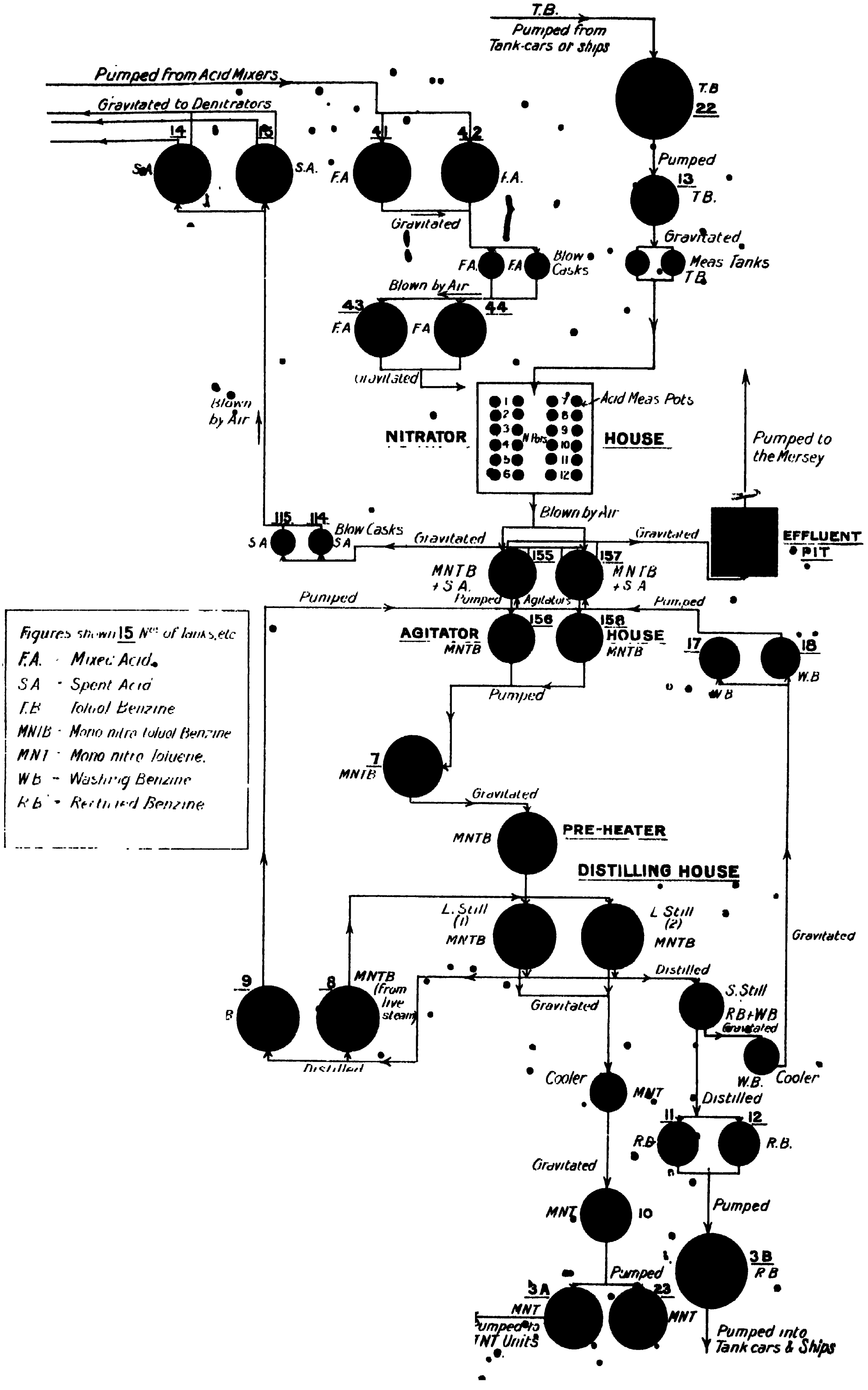
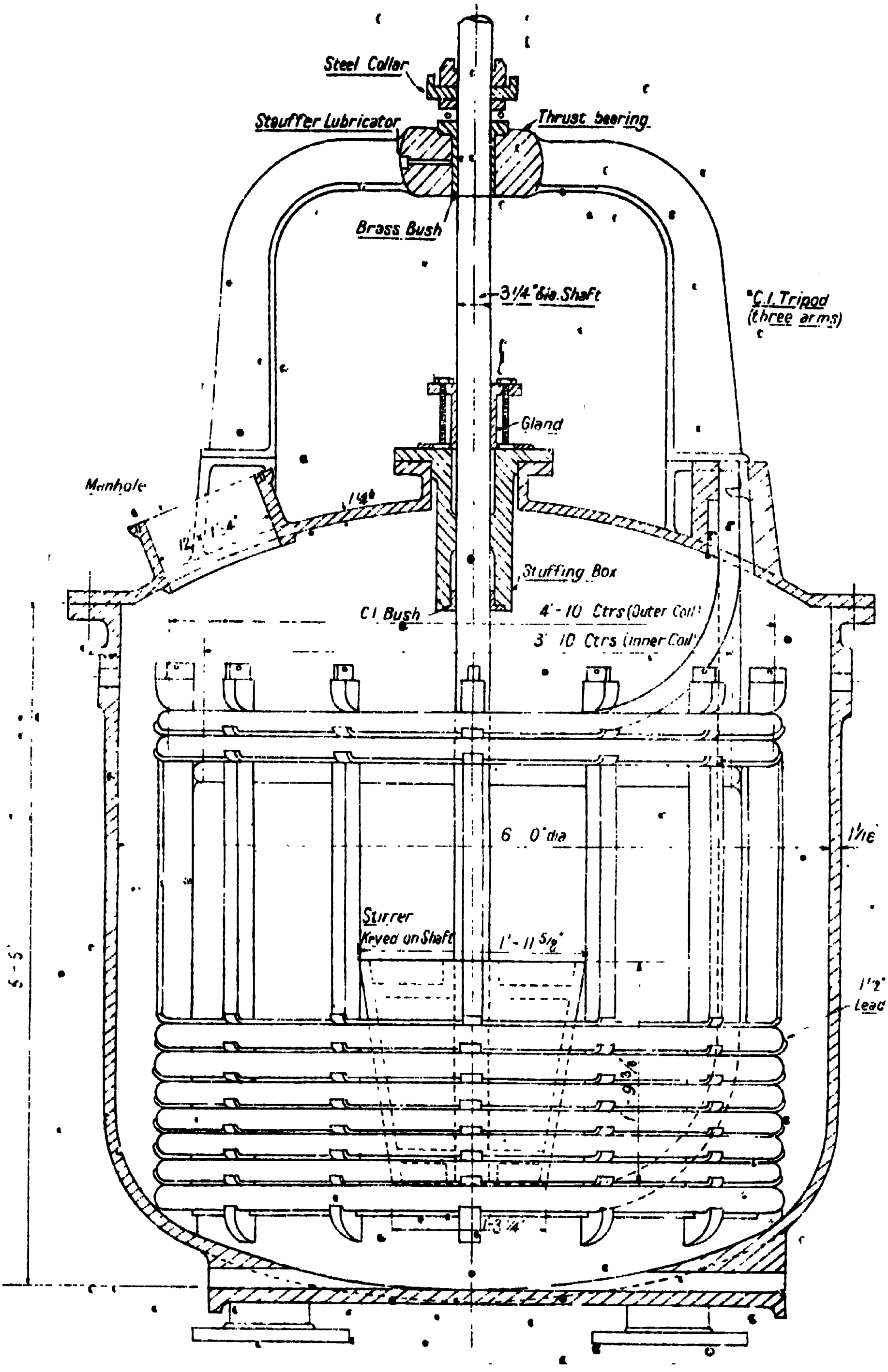
[*] The mixture of MNT and unchanged petrol is known as MNT—B, B being the contraction for the continental term benzine.

[†] See footnote page 27.

Fig. 2.


T.B.
Pumped from Tank-cars or ships
T.B
22
Pumped from Acid Mixers
Gravitated to Denitrators
14
15
S.A
S.A.
41
42
F.A
F.A.
Gravitated
Pumped
13
T.B.
Gravitated
Meas Tanks T.B
F.A
F.A
Blow Casks
Blown by Air
43
44
F.A
F.A
Gravitated
Acid Meas Pots
N Pots
1
2
3
4
5
6
7
8
9
10
11
12
NITRATOR
HOUSE
Blown by Air
Pumped to the Mersey
115
114
S A
S A
Blow Casks
Gravitated
Gravitated
EFFLUENT PIT
155
157
MNTB + S A.
MNTB + S A
Pumped
Agitators
Pumped
Pumped
156
158
AGITATOR
HOUSE
MNTB
MNTB
17
18
W.B
W.B
Pumped
Figures shown 15 Nos of tanks, etc
F.A. - Mixed Acid.
S A - Spent Acid
T.B - Toluol Benzine
MNTB - Mono nitro toluol Benzine
MNT - Mono nitro toluene.
Wb - Washing Benzine
Rb - Refined Benzine
7
MNTB
Gravitated
MNTB
PRE-HEATER
DISTILLING HOUSE
L. Still (1)
MNTB
L Still (2)
MNTB
Gravitated
9
B
8
MNTB (from live steam)
Distilled
Distilled
Gravitated
S. Still
RB+WB
Gravitated
Cooler
MNT
W.B.
Cooler
Distilled
11
12
R.B
R.B.
Gravitated
Pumped
MNT
10
3B
R B
Pumped
3A
23
MNT
MNT
Pumped to TNT Units
Pumped into Tank cars & Ships



— SECTION THRO NITRATING POT — —

Shewing Coils etc in position.

Fig. 3.

(4516) Wt 41844/57 (406) 500 11/20 E&S Ltd to mononitrate in the ordinary way, wash the product free from acid, and distil.

The toluene is received in tank cars from a number of factories in different parts of the country. The nitration is conducted exactly as in the case of toluene benzine. After nitration, the acid is separated as far as possible and the unwashed MNT blown into elevated storage tanks, from which it gravitates as required to the TNT Section.

NITRATION HOUSE AND SUBSIDIARY PLANT.

The nitration house is 75 ft. long x 45 ft. wide, and contains twelve nitrating vessels arranged in two rows of six. Each nitrator is supported on four short iron feet standing on a low brick foundation. The covers and fittings of the pot project through the lead-covered platform which runs along each side of the house 6 ft. 6 in. above the concrete foundation, and from which the nitration is controlled.

Corresponding to each nitrator there is a cylindrical steel acid pot of 2.5 tons capacity. These pots stand on covered stages outside the nitration house, and are raised above the level of the nitration platform so that acid can easily gravitate through the connecting pipe which runs from the measuring pot, under the platform, and up to a funnel in the nitrator cover. On this pipe there is a globe valve next to the measuring pot, and a plug-cock just above the funnel. During nitration the valve is kept full open and the flow of acid is controlled by the cock. The amount of acid is measured by means of a glass float with a stem graduated in ¼-inches, each graduation corresponding to a definite volume of acid. The float consists of a glass bulb, 8 inches in diameter, which is blown on the end of a 6-ft. length of 1-inch tubing and weighted with about 2 lb. of lead shot; a graduated stick is inserted into this stem.

Two toluene-petrol measuring tanks, one corresponding to each row of six nitrators, are situated on raised stages at the end of the building. The charge of toluene-petrol for each nitrator is measured by means of gauge-glasses on the tanks.

Nitrator.—The cast iron nitrators in use are of two types, jacketed and unjacketed. The following are the dimensions of an unjacketed pot (Fig. 3):—

Diameter, 6 ft.
Depth, 5 ft. 5 in.,
Gross volume, 140 cu. ft.
Coils, standards, etc., 15 cu. ft.
Net volume, 125 cu. ft.

The domed cover of the nitrator is of cast iron, and is of sufficient strength to withstand a pressure of 70 lb. per square inch. It is provided with a manhole, thermometer mountings, inlets and outlets for coils, fume pipe, air pipe, discharge pipe, etc., all mountings for which finish on the same level and are provided with slot holes for square-headed bolts. The air pipes lead off from an air main running along each side of the house and connected with a compressed air receiver, in which a pressure of 45 lb. per square inch is maintained by a compressor in the engine house. The discharge pipe inside the vessel is of lead, and is connected with a 3-inch steel pipe running along each side of the house and delivering to the agitators. The manhole can be closed by a cast steel cover held down by two nuts working on eye bolts and bearing on the two lugs at each end of the cover.

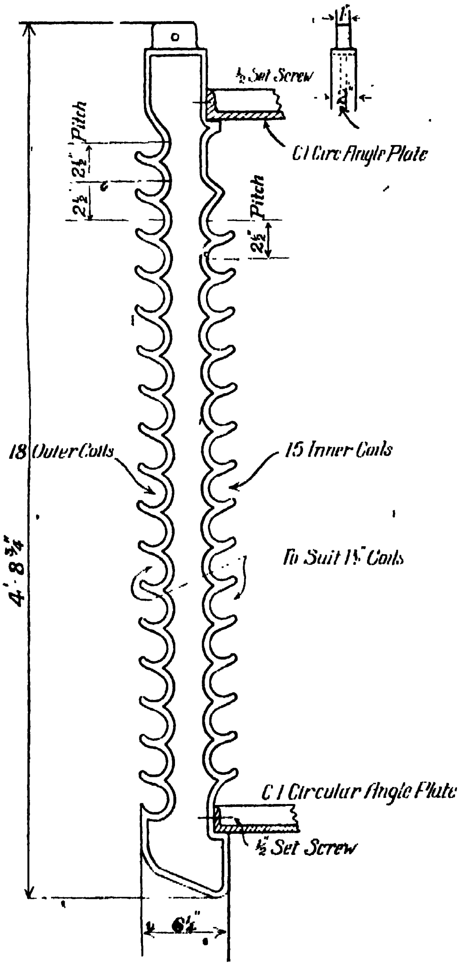
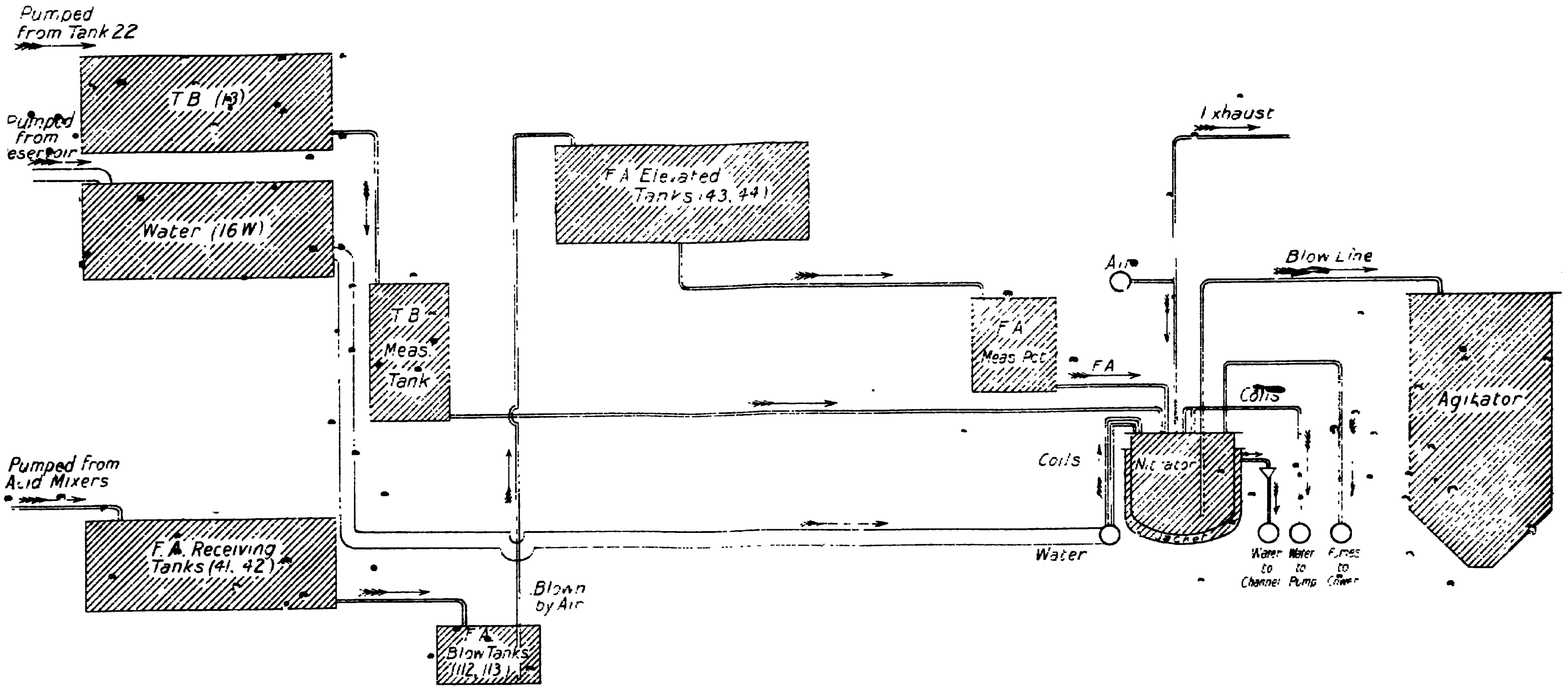


FIG 4.—DETAIL OF DOUBLE COIL SUPPORT.

An asbestos ring in the groove on the main casting enables a tight joint to be made.

When it is found necessary to replace a cooling coil, the latter is lifted in and out of the nitrator by a four-legged sling chain which is attached to the four shackles of a lifting plate, the lifting plate being provided with slots by means of which it is fitted to the coil standards (Fig. 4). The actual lifting is done by travelling cranes running on girders from end to end of the building.



NITRATOR HOUSE.

OPERATIONS.

(Diagrammatic)

The inlets to the two coils are fed from a steel main running along the floor of the house and connected, as shown in the diagram (Fig. 5), with a water storage tank. This tank is supported on four piers, 13 ft. high, and is kept full by pumps in the main water pump house, a constant head being maintained by means of a ball valve in the tank. The coil outlets deliver into one common pipe, in which a vacuum of about 10 inches is maintained by means of two pumps situated in the adjoining engine house. The inlet and outlet of each coil are controlled by gun metal gland cocks.

The water jackets when used are fed from the same main as the coils, but discharge into a separate effluent pipe which leads straight to the drain.

Cooling coils.—Steel coils were originally employed in the nitrating vessels, and though steel, owing to its higher conductivity, is a more efficient cooler than lead, yet owing to the following defects its use has been discontinued. In the first place, with an acid containing approximately 27 per cent. of water (such as MNT spent acid), 35° C. has been found to be a critical temperature for the life of the coils; for, whereas below that temperature the coils last for a long time, above it they are soon eaten through. Secondly, when a coil is thus holed, either water enters the pot, causing rise in temperature and copious evolution of fumes, or acid enters the pumps, according as to whether the coils are under pressure or vacuum. Lead coils on the other hand last for over six months, the first place of attack being in the vicinity of the cast iron lid of the pot. At this point, owing probably to electrical action, corrosion takes place until the coil is holed, but as the hole is above the surface of the liquid in the nitration vessel, and as the coils are worked under vacuum, no water enters the nitrator and no acid enters the pumps.

Stirrer.—Agitation of the contents of the nitrator is carried out by means of a conical "bucket" stirrer of the following dimensions:—

Diameter (top), 1 ft. 11 in.
Diameter (bottom), 1 ft. 3 in.
Height, 1 ft. 9 in.

The bucket (see Fig. 3) is provided with four arms at the top and four at the bottom, both sets of arms being set at an angle of 45°. The whole is keyed to a 3½-inch vertical shaft which passes through a stuffing box in the lid of the nitrator. The weight of the shaft and stirrer is supported by a cast iron tripod bolted to the cover of the nitrator. A 3½-inch Hofmann ball-bearing is provided between the centre bearing of the tripod and a collar on the shaft, the collar itself being secured by a cotter running through the shaft. These bucket stirrers are operated by a 5-inch belt driven off pulleys on the main 3-inch shafting, and they can be stopped or started at will by means of an endless chain operating a rim friction clutch. The bucket stirrer is operated at 146 R.P.M.

Removal of fume.—The fumes evolved during the mononitration, the amount of which is very slight compared with that evolved in the trinitration stage, are drawn away from the pots by means of mild steel pipes connected to a 4-inch earthenware main running along the sides of the building. This fume main leads into a series of earthenware towers, in which a slight suction is maintained by means of an air jet at the top.

Toluene-petrol supply.—The toluene-petrol measuring tanks which supply the nitrators are fed by gravity from an elevated steel tank (No. 13 in the diagram, Fig. 1), which is supported on four brick piers. Toluene-petrol is pumped into this tank as required from the large storage tanks in the tank yard.

The toluene-petrol charge is 1.27 tons, the corresponding figure for coal tar toluene being 0.97 tons.

Mixed acid supply.—When fresh mixed acid is used for the mononitration, it is made up from concentrated sulphuric acid, and nitric acid of about 65 per cent. strength, to the approximate composition—

	Per cent.
H_2SO_4	62.0
HNO_3	20.9
HNO_2	0.2
H_2O	16.9
	100.0

It will be noted that the MNT mixed acid is adjusted to give a residual acid, when the nitration is complete (*i.e.*, when the HNO_3 has been replaced by the equivalent of H_2O), containing about 71 per cent. of H_2SO_4. This strength is chosen in order, on the one hand, that the final spent acid may not be sufficiently dilute to attack the iron vessels in which the nitration is carried out, but on the other hand, so that it may be sufficiently dilute to hold in solution only small quantities of nitro-body, and also sufficiently dilute to avoid even the slightest degree of sulphonation. If the composition can be properly adjusted in this way, the waste acid will be clean and easily separated, it will carry off less than 0.3 per cent. of nitro-body in solution, and after denitration it can be concentrated without frothing or blackening. If, now, on the one hand the waste acid be to any considerable extent weaker than 71 per cent. H_2SO_4, corrosion of the iron vessels will occur, and will generally lead to emulsification of the mixture of nitro-body and acid, apparently by reason of reduction of the nitro-body by the hydrogen formed. If on the other hand, the waste acid is to any considerable extent stronger than corresponds to 71 per cent. H_2SO_4, it will become black and sticky from presence of organic matter, partly in consequence of sulphonation and partly by reason of the solubility of nitro-bodies (more especially of the DNT which is always present in small quantities) in acids which are rich in H_2SO_4.

The fresh mixed acid is pumped to the MNT Section from the main acid plant, over a distance of about half a mile, along a 3-inch steel line, at the rate of 15-20 tons per hour, and delivers into one of two 70-ton steel storage tanks. The 3-inch acid line is supported on a wooden gantry which runs between the Acid and MNT Sections, and is so graded that there is a slight fall from the pump station to the storage tanks. This arrangement serves to keep the pressure on the pipe-line low and minimises the risk of bursts and the amount of leaks. From the storage tanks the acid is raised by means of two 15-ton mild steel blow-casks to two elevated 60-ton tanks situated one on each side of the nitration house. These feed the acid measuring pots by means of a 3-inch line encircling the building.

Nitration process.—The charge of toluene-petrol is run into the nitrators and the stirrers started. The corresponding charge (approximately 2.6 tons) of mixed acid is then run slowly into each pot, about 5 per cent. in excess of the theoretical quantity being used. The temperature is allowed to rise to 35° C. and is then maintained at this figure throughout the nitration by adjusting the flow of cooling water through the coils and the rate at which the acid is added. The rate of addition of the acid is so regulated that the time taken occupies not less than three hours, but in summer, when the temperature of the cooling water may be as high as 25° C. the addition of the acid may require as much as six to eight hours. In order to complete the nitration the contents of the nitrators are stirred for half an hour after all the acid has been added. The specific gravity of the resulting product (MNT-B) in each pot is then determined as a check on the completeness of the nitration. The specific gravity varies between 0.980 and 0.990/15° C., according to the toluene content of the toluene-petrol used. If, owing to faulty working, the specific gravity falls below 0.980, a further quantity of mixed acid is added. If all the gravities are correct, the contents of the nitrators are cooled to 30° C., the manhole lids are screwed on, and the total charge of six pots blown over by compressed air into one of the lead-lined agitators in the adjoining house. The time required to discharge the contents of one nitrator is about 5 minutes, and by means of an electric bell the moment of despatch, arrival, etc., are signalled from one building to the other.

When coal tar toluene is used instead of toluene-petrol the acid charge is approximately 3.2 tons, the time taken to add the acid being in this case about one hour longer than in the former; the final specific gravity of the product is about 1.165. After nitration is complete, the contents of the six pots are blown over as described above, but in this case they may be received into any of the four agitators.

ENGINE ROOM.

This building, which is 41 feet square and separated from the nitration house by a narrow alley way, contains the engine for driving the stirrers in the twelve nitrators. One engine of about 60 H.P. is required for this purpose. The engine house also contains two suction pumps for drawing the cooling water through the nitrator coils, and two air compressors which are arranged so as to cut out automatically when the pressure of air in the receivers reaches 45 lb. per square inch.

AGITATOR HOUSE.

The agitator house is a two-storied building, 37 ft. × 30 ft., and the adjoining engine house, which is a lean-to on the main building, is 20 ft.

square. The two stories are connected by means of a spiral staircase situated in the centre of the building. Suspended from the girders supporting the upper floor and with the greater part of their bodies below it, are four cylindrical washers or agitators, built up in sections of ⅜-inch mild steel plates. The lower ends of these vessels consist of shallow cones terminating in a plate 2 ft. 6 in. in diameter. The vessels are each 15 ft. long overall, 8 ft. 6 in. in diameter, and of 22,000 litres capacity. The top covers are flat and carry mountings for the MNT-B, water, and light benzine pipe-lines, etc. To the covers are also fixed the cast iron tripods which take the weight of the central shaft and stirrers in the same way as in the case of the nitration vessels. The stirrer shaft is further steadied by a stuffing-box where it passes through the cover, and at the bottom end by a footstep bearing. A cast iron shroud shrunk on to the steel shaft works in the cast iron footstep, the latter being grooved spirally and kept flushed out through a one-inch drain line during the washing process. This method of keeping the bearing clean is found to be more satisfactory than attempting to exclude the acid, etc., altogether. The cover of the agitator is provided with a large manhole with a swing lid.

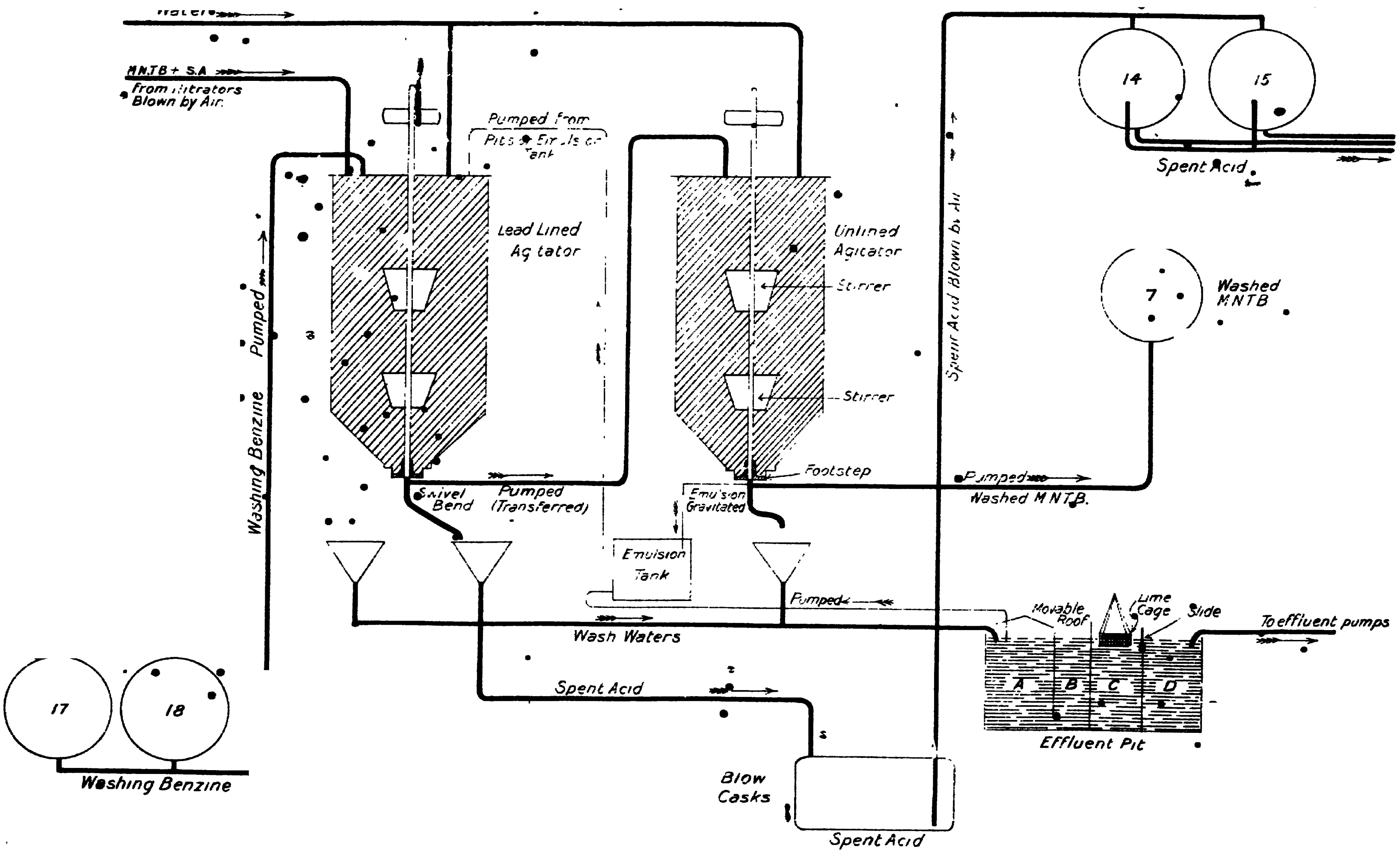
From the top floor are controlled the various valves and cocks on the pipe-lines delivering into the agitators, and also the clutches which operate the stirrers. The bucket stirrers are similar to those used in the nitrators, except that two buckets and one pair of disrupter blades are fitted to each stirrer shaft (see diagram, Fig. 6).

The operation of running off material from the washers, and the manipulation of all exit pipes and valves are controlled from the bottom floor.

Two of the agitators are lead-lined (½-inch lead on bottom, ⅜-inch on walls, and 3/16-in. on stirrer shaft and buckets), and are connected with the discharge line from the nitrating vessels. In these agitators the spent acid is separated off and the MNT-B washed neutral. The remaining two agitators are not lead-lined and are only brought into use after the MNT-B is neutral. The 3-inch outlet pipe from the lead-lined agitator is attached eccentrically to the bottom plate, and in the case of the other agitators it is closed by means of a 3 inch gate valve which is connected to a ⊥-piece. One arm of the latter leads through a valve on the delivery line to an MNT-B storage tank, and the other is connected by means of a cast iron stand pipe to the 6-inch cast iron effluent main (Fig. 6).

In the case of the lead-lined agitators the outlet is connected to one arm of a cast iron ⊣-piece, the side opening of which leads through a 3-inch regulus metal valve to the transfer line used in pumping the contents of a lead-lined into an un-lined agitator. The lower opening leads through a regulus valve to a swivel pipe, which can be swung so as to deliver into one or other of the two lead-lined funnels. One of the latter is situated below each of the lead-lined agitators and is connected by a 4-inch cast iron pipe to two 15-ton steel blow-casks into which the spent acid is run after separation from the MNT-B, and from which it is blown into two elevated steel tanks from which it gravitates to the Acids Section to undergo denitration and concentration. A certain amount of MNT-B separates from this spent acid on standing and accumulates on the surface of the acid in the storage tanks The MNT-B which separates is periodically returned to the agitator

M.N.T.B. WASHING.



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by a skimming pipe, and is rewashed. The second funnel is connected to the effluent main referred to above, which delivers into one of three effluent pits.

Procedure in the case of MNT manufactured from coal tar toluene.—If coal tar toluene has been used, the contents of the nitrator are blown direct to settling vessels, from which the acid is drawn off and the MNT delivered direct to storage. In this case the discharge line from the nitrators is connected to all four agitators, each of the latter having an acid run-off and a connection to an MNT blow-cask. The agitators in this instance are used merely as separators, and, after the MNT and acid have been blown over from the nitrators, the contents are allowed to settle for about four hours, after which time the acid is separated.

The unwashed MNT has a specific gravity of approximately 1.165/15° C., and a flash point in a semi-enclosed apparatus of about 70° C. When fresh mixed acid is used for the nitration, the spent acid produced has approximately the following composition :—

	Per Cent.
H_2SO_4	72.0
HNO_3	0.3
HNO_2	0.4
H_2O	27.3
	100.0

When all the acid has been separated, the MNT is run off through the side connection into the blow cask and blown up into elevated steel storage tanks, from which it gravitates to the TNT Section. The product contains about 0.5 per cent. of un-nitrated toluene, and approximately 0.5 per cent. of paraffins, mainly octanes.

Procedure in the case of MNT manufactured from toluene-petrol.—After the contents of the six nitrating pots have been received into a lead-lined agitator they are allowed to settle for at least half an hour; the longer the period of settling allowed before separation, the easier is the subsequent washing of the MNT-B. The spent acid is then run off as completely as possible. A sample of the spent acid from each nitration is taken for analysis, and it is by the nature and content of the spent acid that the amount of fresh acid to be used is adjusted. An average analysis of the spent acid is given in the preceding paragraph.

Any un-nitrated toluene is lost during the subsequent distillation process, and in order that this loss shall be as small as possible the charge of toluene-petrol is calculated so that it will yield a spent acid containing approximately 0.4 per cent. of nitric acid. The MNT-B, when drained as completely as possible, holds about 0.5 per cent. of nitric acid in solution but contains practically no sulphuric acid.

Washing of the MNT-B.—When MNT is manufactured from toluene-petrol it is necessary to separate the unchanged petrol from the MNT-B by distillation, and for this purpose the mixture must first be freed completely from acid.

The operation of washing is not altogether simple, on account of the fact that the specific gravity of the mixture of MNT and unchanged petrol, of the composition generally obtained in the nitration, is very near to 1.0. In consequence it may undergo emulsification, and the liquid may refuse to separate. This is especially liable to occur in the alkali wash, owing to the tendency of petrol to emulsify with alkaline liquids. Where difficulty is experienced in the washing by refusal of the nitrated mixture to separate from the wash-water, it is customary to add some of the petrol recovered by distillation from previous charges. The specific gravity of this liquid is about 0.770, and addition of a suitable quantity will, therefore, ensure satisfactory separation.

Before washing the acid MNT-B remaining in the agitator, therefore, its specific gravity is lowered to 0.945-0.940 by the addition of 2.3 tons of petrol residues (see page 44). Traces of acid are once again drained off, and the stirrer started. Water is then sprayed into the MNT-B from a perforated 3-inch lead pipe which encircles the top of the agitator. After sufficient water has been run in, the stirrer is stopped and the inflowing water is allowed to fall through the MNT-B layer, the acid water being run off simultaneously from the bottom. This continuous water wash is allowed to go on for about five minutes, after which time the inflowing water is turned off and the water remaining in the agitator drained. This operation removes the bulk of the acid, but as a rule three or four further washes are required in order to render the MNT-B neutral. In carrying out the later washes, water is run in with stirring until the agitator is nearly full. The water feed and stirrer are then stopped, and after five minutes settling the water is run off. About 2 tons of water are used for the continuous wash and about 6 tons for each of the subsequent washes. When the last drainings of wash-water are neutral to litmus, the batch of MNT-B is pumped over to another agitator (not lead-lined) in which it originally received a wash with dilute caustic soda, but this latter operation has been omitted for some time. The purpose of the alkali wash was to remove nitro-cresols, but it has been shown that the latter do not cause trouble in the subsequent nitration stages and the alkali wash was therefore discontinued.

As a precautionary measure, however, against the possibility of allowing any acid to reach the stills, the MNT-B is given two additional washes, using in each case about 4 tons of water. It is then allowed to settle for half an hour and any water that has separated is drained off, together with the small amount of emulsion which tends to accumulate below the layer of MNT-B. This emulsion is run into a small steel tank from which it is pumped at intervals back into a lead-lined agitator where it is mixed with a charge of acid MNT-B from the nitrators. It is found that the emulsion does not accumulate when treated in this manner.

The clean, neutral MNT-B is then discharged to a tank which acts as a storage vessel for the large still.

EFFLUENT PIT.

The wash waters from the agitators are conveyed by means of a lead-lined launder into one of three effluent pits. These pits consist of four compartments, as shown in Fig. 7. The wash waters, together with a certain unavoidable quantity of MNT-B, flow into the covered receiving pit A. This pit is 19 ft. × 10 ft. × 6 ft. 6 in. deep, and in it the light MNT-B separates out on the surface, whilst the water flows on under the dividing wall into section B, and thence into the neutralising pit C. The latter is kept well agitated by means of compressed air, and the acid water is here neutralised with lime which is introduced into the pit in an iron cage suspended by a tackle from a girder running overhead. When the contents of C are neutral, the movable slide which separates this pit from the fourth compartment is raised and the effluent waters flow into D, from which they are pumped to the TNT Section where they mix with the treated TNT effluent.

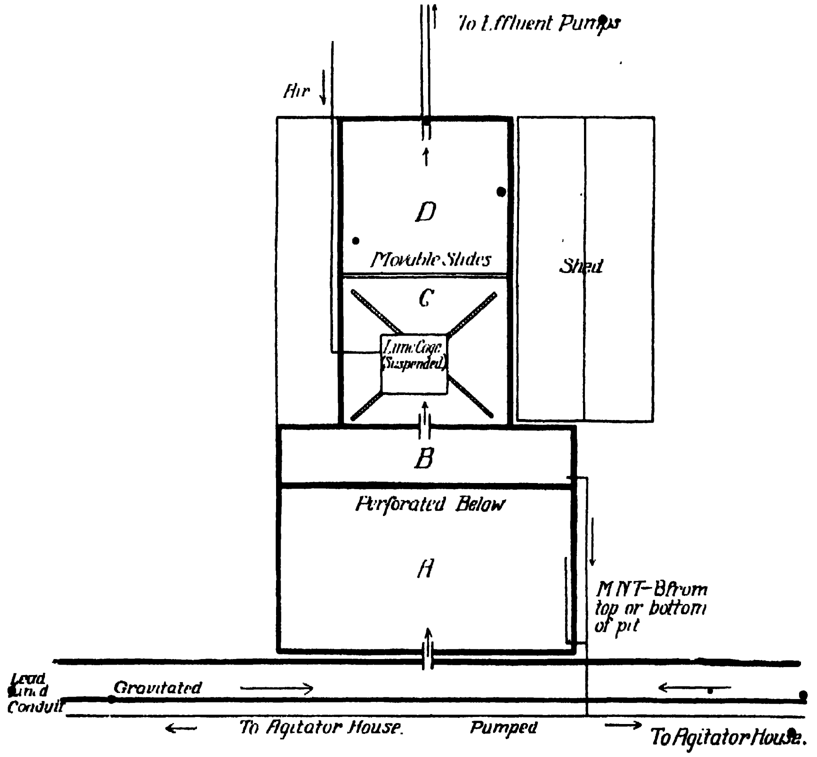


FIG. 7.—EFFLUENT PIT.

The MNT-B which separates in A and B is removed by means of an adjustable skimming device from the surface of the receiving pit, or, if much evaporation has occurred and the MNT-B in consequence has sunk, from the bottom of the pit; the material so recovered is re-washed in a lead-lined agitator. A few buckets of spent acid added to the agitator facilitates this operation, as it assists in coagulating the emulsion. The effluent pits are built of blue-brick and cement and are double walled, the space between the walls being filled with asphalt.

PUMP HOUSE.

This building contains eleven pumps for pumping the various liquids in use; three process pumps for filling stills, emptying agitators, etc., two for MNT, one large and one small pump for toluene-petrol, one for toluene, and two for petrol. All except the process pumps are arranged so as to pump to and from the tank cars in the siding alongside the pump house.

THERMAL STORAGE TANK.

The duty of this tank, which is a converted 30 ft. × 8 ft. 6 in. Lancashire boiler (Fig. 8), is to store all exhaust steam from the plant for use as required. Exhaust steam is led from all engines, compressors, and pumps to a 6-inch header extending the entire length of the tank and as high up as possible. Forty-eight 1-inch branches are taken from this header, each branch being fitted with a cast iron nozzle in the shape of a cross. Each nozzle is placed inside a 6-inch petticoat pipe and the nozzle itself is about 4 inches below the surface of the water in the tank. The object of the petticoat pipe is to bring about good circulation of the water, and all such pipes are given a slight inclination towards one end of the tank where the blow-off is fitted. This is done in order to prevent scum and oil remaining on the surface of the water, which would retard the working of the tank. The blow-off works automatically or not, as desired, and consists of a float valve at the base of a funnel, the float closing or opening the valve so as to maintain a constant water level. The funnel is to assist in removing the scum and oil from the tank. Other fittings consist of a 7-inch safety valve operating at 7 lb. per sq. in., by-pass valves for steam inlet, and small water sprays. The water supply to these sprays, the object of which is to keep the tank pressure below 7 lb. per square inch, is automatically controlled by the steam pressure in the tank. When the pressure rises above 6 lb. cold water is sprinkled through the sprays all over the surface of water in the tank. All excess of water is blown off through the funnel to a filter and is from there pumped back to the boilers.

The whole arrangement is well insulated, and supplies sufficient steam for heating the nitrating house, the agitator house and the pre heater in the distillery, and a moderate though uncertain supply of hot water for the boilers.

DISTILLERY.

The MNT-B, now free from acid, is distilled by means of superheated steam from a still fitted with a small refining column. The first fractions are pure petrol, but after distillation has been in progress for some time, the petrol carries over small quantities of MNT. These intermediate fractions must be collected separately for subsequent distillation. The residue in the still is MNT free from petrol, and after cooling it is delivered to storage tanks. The intermediate fractions are re-distilled in a still fitted with a large refining column, and separation of the petrol from MNT is made almost complete.

The recovered petrol, being of almost constant composition and properties, finds numerous applications. It is especially suitable for extractions of various oil seeds, and is largely used for this purpose as well as for the cleaning of various delicate fabrics and similar purposes. It is

To face p. 70.

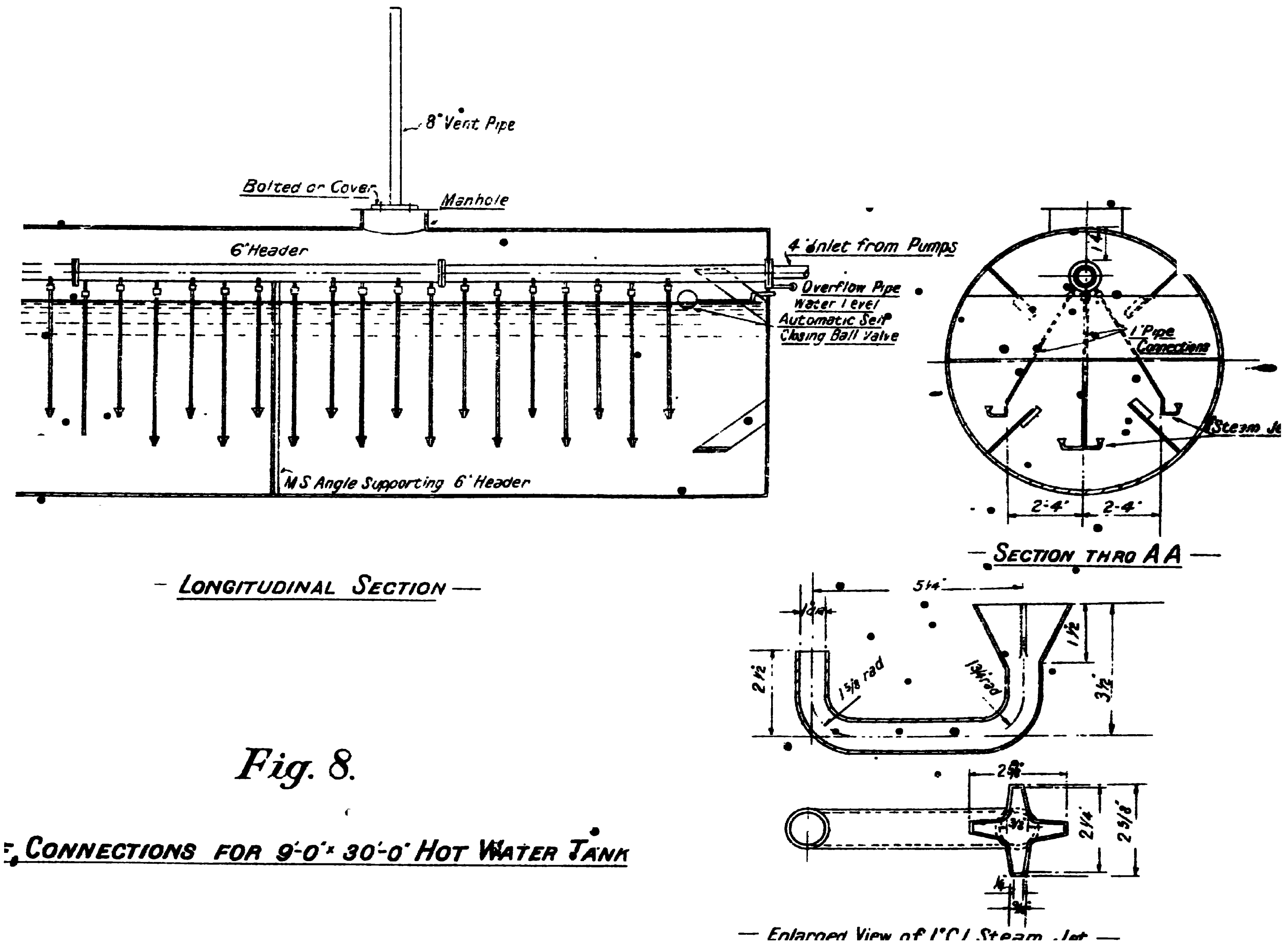


Fig. 8.

CONNECTIONS FOR 9'-0"× 30'-0" HOT WATER TANK

generally not absolutely free from MNT, and traces of this material have been detected in seed-cake used for cattle food, which has been obtained after extraction of oil-seed by means of the recovered petrol.

Stills.—The still bases consist of mild steel riveted shells of the following dimensions:—

Large Still.—9 ft. 2¼ in. diameter × 25 ft. long × ⅜ in. thick, having a capacity of about 1,700 cu. ft.

Small Still.—6 ft. 6¾ in. diameter × 22 ft. long × ⅜ in. thick, having a capacity of about 700 cu. ft.

Each still is provided with a Heckmann column which consists of a series of trays one above the other. Each tray is fitted with 10 hoods and 4 drop pipes, shown diagrammatically in Fig. 9. The large stills have short columns containing eight trays, and the small stills long columns carrying 27 trays. The petrol vapours rise through the raised hoods on each tray to the top of the column, passing through the layer of liquid condensed in the tray and maintaining it in a constant state of ebullition, while the excess of liquid on the tray overflows through drop pipes which discharge into the tray immediately below. Finally, the vapours reach the condenser (see later) where they are condensed to the liquid form. In the case of pure rectified petrol, the condensed liquid passes through a small water separator and then through a sight-glass by means of which the rate of distillation may be controlled. The sight-glass consists of a cylindrical glass vessel surrounding an inner vessel with a perforation at its base (Fig. 10). The distillate passes upwards in the inner glass and escapes through the perforation into the annular space between the two glasses, flowing thence through a 3-inch pipe to the receiving tank outside the still house.

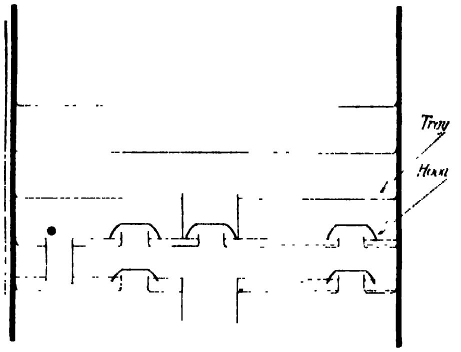


FIG. 9.—VERTICAL SECTION OF COLUMN OF LARGE STILL.

The stills are heated by four 1½-inch horizontal lap-welded steam coils, into which steam can be led at 160 lb. pressure. In addition to the above, a "live" steam coil passes along the bottom inside the large stills. Both sizes of stills are fitted with relief valves, pressure gauges, and gauge glasses. The still residues are released from below, through a 4-inch valve in the still base, to coolers.

MNT coolers.—The coolers are mild-steel riveted shells, 19 ft. 9 in. long × 4 ft. in diameter, containing 180 18 ft. × 2 in. steel tubes provided with double tube plates. The MNT enters the coolers at the top, passes outside the tubes to the end, and returns, leaving the cooler at the bottom outlet, directly opposite to the inlet. The tubes are water-cooled, the water flowing in a direction opposite to that of the MNT.

Condensers.—These consist of vertical steel shells, the larger containing 222 tubes, $1\frac{1}{2}$ in. diameter × 13 ft. 7 in. long, the smaller containing 129 tubes $1\frac{1}{2}$ in. diameter × 13 ft. 7 in. long; all tubes are of solid-drawn steel. The cooling surface of the large condenser is 1,189 sq. ft., and that of the small condenser 694 sq. ft. The petrol passes into the condenser at the top and on the outside of the tubes. The cooling water, which is supplied by means of a 3-inch pipe at the bottom, passes up through the tubes and overflows at the top.

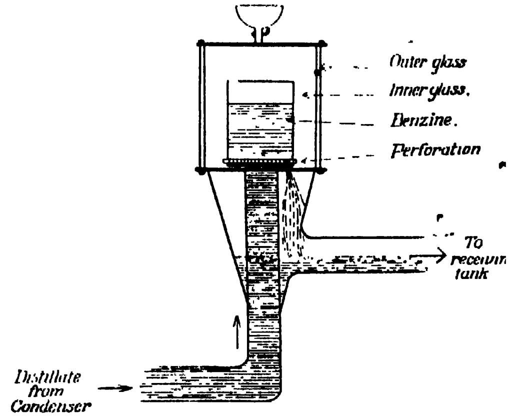
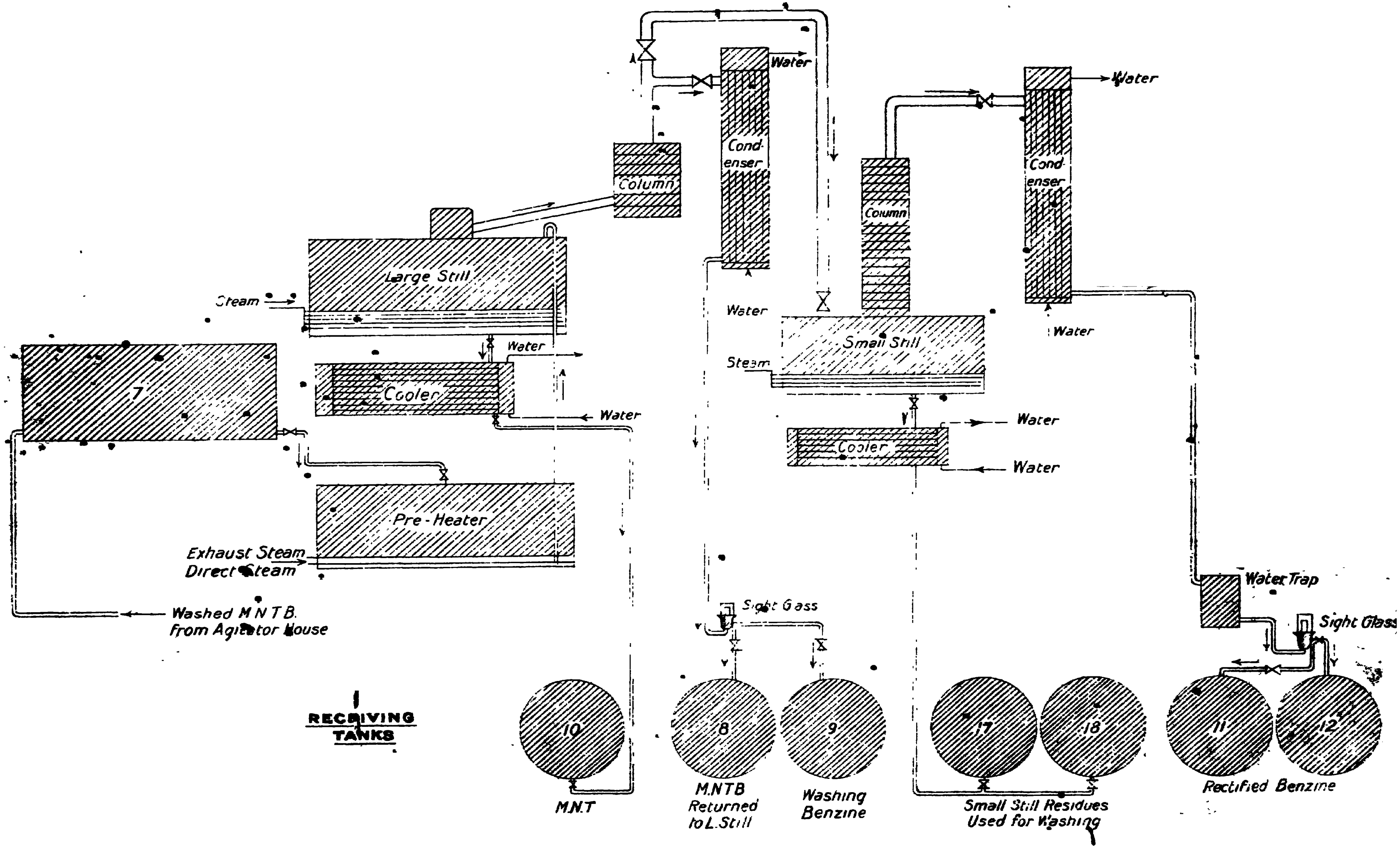


FIG. 10.—SIGHT GLASS.

Preheater.—The preheater consists of a converted boiler, 28 ft. long × 8 ft. 6 in. diameter × $\frac{5}{8}$ in. thick, and of approximately 1,500 cu. ft. capacity. The exhaust steam from all engines, pumps, compressors, etc., is led into the thermal storage tank described on page 40, and thence supplied to the preheater at a pressure of about 7 lb. per sq. in. The steam inlet is a 6-inch wrought iron pipe which supplies ten 3-inch wrought-iron horizontal steam coils; there are in addition four $1\frac{1}{2}$ in. wrought iron high pressure steam coils. The preheater is furnished with mountings for a dead weight safety valve, 3-inch discharge pipe to the still, 3-inch filling pipe, 3-inch vapour pipe, gauge glasses and thermometer. The vapour pipe and exhaust from the safety valve are led to the condenser.

Distillation process.—After the water has been drained off the MNT-B in the elevated storage tank (No. 7, Fig. 11), the preheater is filled to within a few inches of the top. Exhaust steam is turned on and the temperature rises to about 80° C., further water being drained off at the same time. Direct steam is next applied, and in about $2\frac{1}{2}$ hours the pressure in the pre-

DISTILLING HOUSE.



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heater rises to 10 lb. per sq. in. at a temperature of 109°-110° C. As the capacity of the preheater is less than that of the still, it is necessary to pump 4 or 5 inches (measured on the gauge glass) into the still before blowing up the main bulk of the MNT-B. The latter process occupies 75 minutes and is accompanied by the appearance in the sight-glass of a certain amount of distillate, owing to the temperature of the liquid. Heat is now applied to the still by means of its own coils, and at about 105° C. distillation commences.

The first distillate generally contains water and has a high specific gravity (0.755); it is therefore run for fifteen minutes into a fraction tank (No. 8, Fig. 11). The distillate is diverted into another fraction tank during the next hour (No. 9, Fig. 11), and by the end of that period a steady flow of water-free petrol is obtained, having a specific gravity of 0.747. The valves at the top of the column are adjusted so as to turn the distillate into the small still, which as a rule already contains a small amount of petrol from a previous operation. Should the temperature of this be too low, the steam coils in the small still are turned on. By this means the distillate appears in the sight glass sooner than would have been the case had the large still distillate been turned into a cold still.

Distillate.—Continuous distillation proceeds for about 7½ hours, the product ("rectified benzine") of gravity 0.748 at 15° C. being run to special fraction tanks (Nos. 11 and 12, Fig. 11). Samples are taken at the end of the first hour, and then after every two hours, and these are tested in the laboratory for final boiling point. At the commencement of operations this boiling point is 117° C., but if it rises to 118.5° C. samples are taken hourly, whilst a final boiling point of 119° C. necessitates the cessation of continuous distillation, or alternatively the small still is nearly emptied by gravitation into tank 17 or 18. The distillate is then diverted from the small still and passed through the large still sight-glass into a fraction tank (No. 9, Fig. 11) for about an hour. By the end of this time all the steam coils have been fully turned on, and yet the sight glass is not full as it has been hitherto. In other words distillation fails, and it is necessary to resort to the use of "live" steam in order to drive off the rest of the petrol. Directly live steam is admitted to the still the product is diverted to another fraction tank (No. 8, Fig. 11) and the gravity rises rapidly from 0.765 upwards. The steam causes MNT-B and water to pass over, in which the proportion of petrol diminishes continuously. The gravity consequently rises ~~until~~ in about an hour it becomes greater than 1.0. This stage is called the "reverse" owing to the fact that a brownish layer of water appears in the sight glass, superimposed on the greenish yellow MNT-B. Half an hour after this a sample is withdrawn from the gauge-glass at the back of the still. This sample is distilled in the laboratory and the first few drops tested for petrol by a flash test. If petrol is absent the steam is shut off, and the residual MNT is run into the coolers described above and finally to a special fraction tank (No. 10, Fig. 11).

During the passage of live steam, the temperature of the still rises from 164° to 174° C. The temperature of the water in the condenser is about 25° C. throughout the greater part of the distillation, but rises towards the end to 30° C. or more.

As there are two large stills in the unit here described, directly one is turned off from the small still the other is as a rule ready to turn into it, so that the small still is continually in use, being emptied (usually to six inches in the gauge-glass) either because it becomes inconveniently full or because the final boiling point rises above 119° C. It is this small still residue which is employed in reducing the specific gravity of the MNT-B in the agitators (see page 38).

TRINITRATION.

General description of TNT section.—The general lay-out of this Section is shown in Figs. 12 and 13. It consists of 5 separate units (A., B., C., D, and E., Fig. 12), distanced 100 yards apart, each of which comprises:—

Nitration House, containing 4 nitrators, 4 separators, 4 detoluators, etc Adjacent to the nitration building are absorption towers and fanhouse, storage tanks for MNT, mixed and spent acids, etc., and pump house containing the necessary pumps for elevating the MNT and acid into the nitration plant. The crude TNT is conveyed in the solid state from the nitration house to the

Washing House, in which the TNT is washed, dried and granulated, and from which it passes to the

Packing House, in which the granulated TNT is packed in boxes which are stored in the

Magazine, to await despatch.

In addition to these 5 units there are the following subsidiary plants:—

Sulphite Dissolving House, in connection with those units operating the sulphite washing process.

Box Factory, for the manufacture of new boxes.

Box Store, in which returned empty boxes are stored, examined and prepared for re-issue.

Liner House, in which all returned box-liners or bags are sorted and shaken to remove any TNT which they may contain.

Recovery Plant, for treating TNT which has either been contaminated with dirt, etc., or recovered from the wash-water launders and settling pits.

Steaming Plant, at which portable parts of the plant are steamed and cleaned before undergoing repair.

Lead Burning Plant, where scrap lead, after being thoroughly cleaned at the steaming plant, is melted and cast into pigs ready for sale.

Effluent Plant, for receiving, neutralising, and pumping away the wash waters leaving the nitration and wash houses.

General outline of trinitration process.—As operated latterly, the process (*i.e.*, including mononitration) has been practically a three-stage one. The general details can be followed out in the diagram, Fig. 14, which shows the nitration house in diagrammatic elevation.

A charge of mixed acid is blown up by compressed air into the nitrator and is there used to complete the nitration of the charge of nitro-body separated from the detoluator, which has been blown up from an egg, through

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FIG. 12.

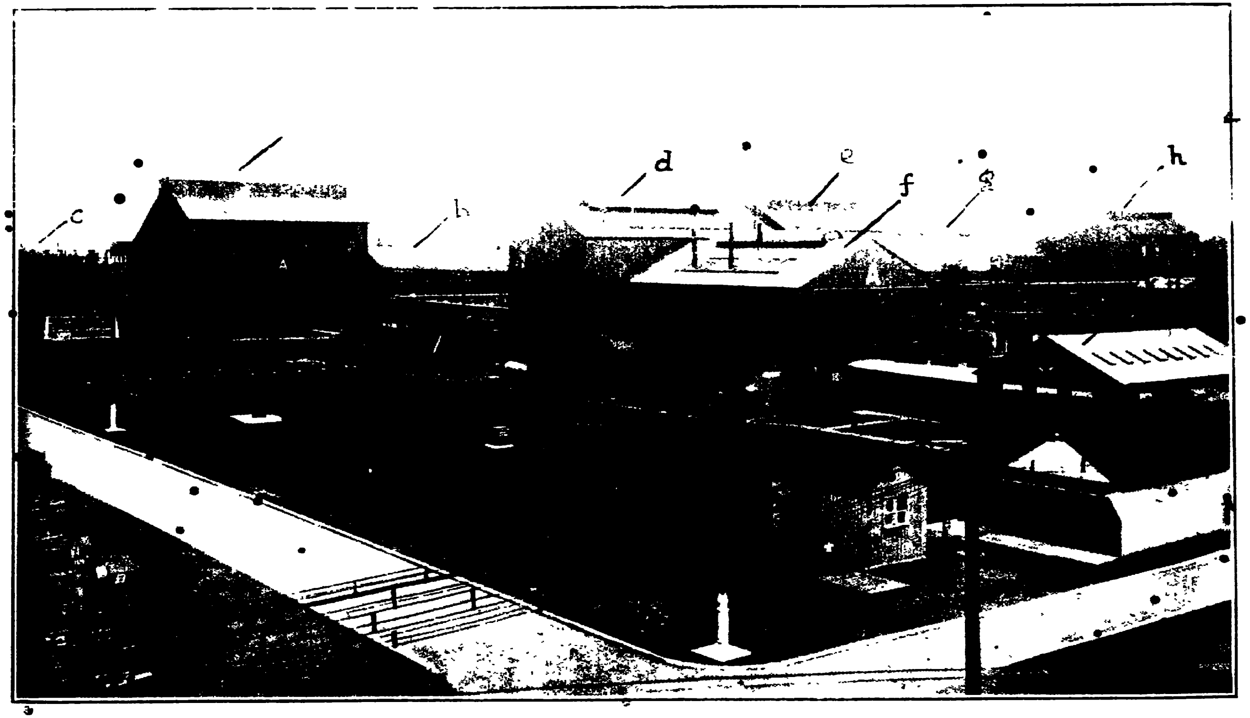

FIG. 13.

a. "A." nitration house.
b. Boiler house: TNT section.
c. "A," fume towers.
d. "A." wash house.
e. "B." nitration house.
f. "A." drying and granulating house.
g. Reservoirs for river water.
h. "C," nitration house.
k. "A," packing house.
l. "A." granulator fan house.
m. "A." granulator motor house.
n. "A," motor house for washing gear.

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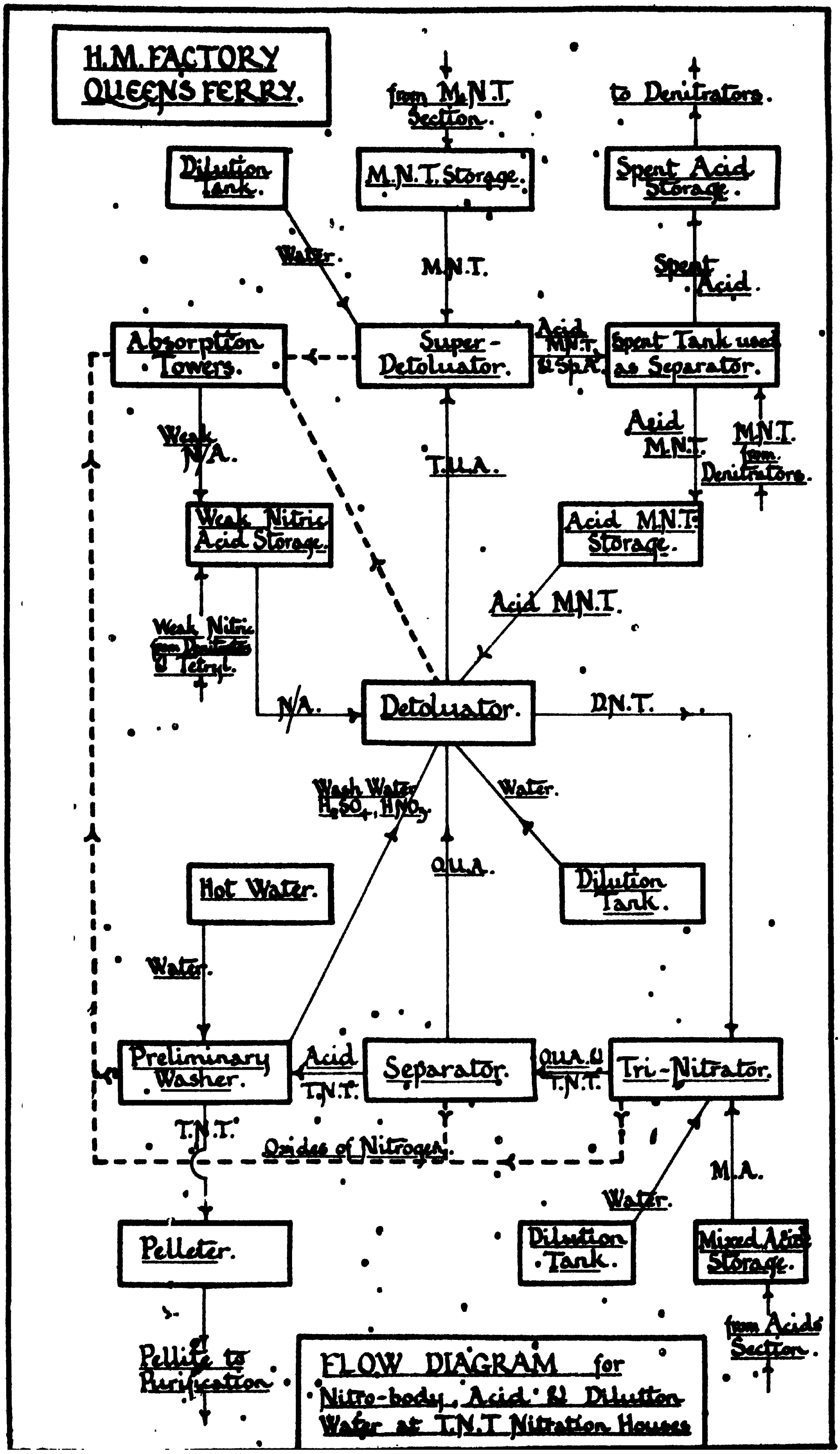

H.M. FACTORY QUEENSFERRY.

FLOW DIAGRAM for Nitro-body, Acid & Dilution Water at T.N.T Nitration Houses a splash tank, to a storage tank feeding the nitrator. This nitro-body (hereafter referred to as "DNT") consists mainly of dinitrotoluene, sufficient weak nitric acid having been added to the detoluator (which thus becomes a dinitrator) almost to complete the dinitration of the MNT. When trinitration is complete, and after adequate dilution with water (which is necessary to secure proper separation of the nitro-compounds), the whole charge of TNT and acid (once used acid, or "O.U.A.") is run to a separator. After standing for about an hour, the once used acid is run to the detoluator and the charge of approximately 1 ton of TNT is run into the preliminary washer, where it receives a wash with 400 lb. of boiling water; this water is afterwards used in the detoluation stage for dilution purposes. The TNT is then "pelleted" by means of cold water and conveyed to the wash houses in this form for final treatment.

To the once used acid in the detoluator is added a measured charge of so-called acid-MNT (which has resulted from a previous super-detoluation stage), followed by a charge of weak nitric-acid.

After adequate stirring, the water from the preliminary washer is added, together with any further dilution water which may be necessary at this stage. The charge is then cooled and allowed to stand for separation to take place, after which the acid (twice used acid, or "T.U.A.") is run to a super-detoluator, while the nitro-body is run down to an egg and blown up to the DNT tank ready for feeding into the nitrator.

A charge of fresh MNT is then run into the twice used acid in the super-detoluator, together with the final fraction of the dilution water. After thorough cooling, the whole charge of spent acid and nitro-body (MNT containing a small proportion of DNT in solution) is run into one of the spent acid tanks which is used as a separator. After allowing a sufficient period to elapse for separation to take place, the acid MNT is skimmed off into the acid-MNT tank which supplies the nitro-body charge for the detoluators, whilst the spent acid is siphoned into a second spent acid tank, where the final separation takes place, after which the acid is pumped away to the denitration plant on the acids section.

Fume mains from nitrators, separators, preliminary washers, detoluators and super-detoluators are led into a battery of absorption towers, in which the oxides of nitrogen are recovered as weak nitric acid which is used in the nitric acid charge for the detoluators.

A flow-diagram for the process, as described above, is shown in Fig. 15 on the opposite page.

In the detailed account which follows the plant will be considered prior to the process.

STORAGE FOR ACIDS, MNT, ETC.

Storage accommodation is provided on each unit for mixed acid, spent acid, MNT, and acid-MNT from the super-detoluators.

Mixed acid.—Two cylindrical storage tanks (a, Fig. 16), 20 ft. diameter × 12 ft. high, constructed of ⅜-inch mild steel boiler plates, are provided at each nitration house. A 2-inch connection, for levelling the acid in the two tanks, is fitted at a height of 11 ft., giving a working capacity of 345 cu. ft. or 177 tons for each tank, which is sufficient to permit the tanks to be used on alternate days for the receipt and issue of mixed acid. Each tank is fitted with a 20-inch diameter manhole at the centre of the cover, this being removed for cleaning purposes. The mixed acid is delivered into the centre of the manhole through a 2½-inch delivery pipe. The main outlet from the tank is 3 inches in diameter, and is fitted with a seating projecting 3 inches above the bottom of the tank to permit the accumulation of sludge. Into this seating fits a regulus metal plug, 4½ inches long and tapering from 5¾-3 inches, which is raised and lowered by an attached rod. The main object of the plug is to take the weight of the acid off the outlet cock, and to serve as a standby in case of accident to the outlet connections. The swivel attachment at the top of the plug-rod is disconnected daily and the plug rotated in its seating. This has very considerably increased the efficiency of the plug, in so far as it prevents the deposition of a hard cake of sludge on both plug and seating.

The bottom of the tank is provided with a 2½-inch drain outlet, fitted with a plug-cock. This is used for sludging, or for drawing off the acid in case of accident to the main outlet connection. The tanks are filled from the acid mixing plant by a 4-inch main with branches to each TNT unit. Acid is received at the rate of 40 tons per hour, which enables a day's supply for all units to be received in 12 hours.

MNT.—The storage for MNT comprises 2 tanks (b, Fig. 16) at each nitration house. Each of these consists of a mild steel boiler shell, 19 ft. 6 in. long × 6 ft. diameter, of 3,400 gallons capacity. The tanks are built up of ½-inch mild steel plates, fitted with one 2-inch outlet saddle at the bottom; three 2-inch saddles on top; one 2½-inch outlet for use in the event of it being necessary to use the tank as a blow cask; one 1-inch air inlet for the same purpose; and a 16-inch manhole frame and cover. Each pair of tanks is built within four brick walls, on a bottom of tarred bricks, so that in the event of leakage the MNT is confined and may afterwards be recovered through a 2-inch pipe which passes through a hole in the wall at the lowest corner of the enclosure. Several of these tanks have since been inverted, as the acid which separated out from the "unwashed" MNT (see page 27) attacked and perforated the bottom of the tanks. The outlet elbows which were made originally in wrought iron were also rapidly corroded, and have been satisfactorily replaced by cast iron. In fact, cast iron or lead pipes are essential for use with all nitro-bodies which have been imperfectly separated from acid.

During the cold winter of 1917, there was also considerable trouble and delay due to the freezing out of the meta-isomer in the MNT, which settled as a sludge in the bottom of the MNT tanks and suction lines to the pumps. The pipe-lines had to be frequently steamed, and to obviate the difficulty steam coils were installed in some of the MNT storage tanks.

Spent acid.*—Each nitration house is provided with two lead-lined storage tanks for spent acid (c, Fig. 16), each 20 ft. × 12 ft. × 7 ft. 6 in.

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* A more detailed description and drawing of this plant is given in the first publication of this series, viz., "Denitration of Spent Acids."

FIG. 16.

To face p. 48.]

deep, and with a fall of two inches towards the outlet end. All inside angles are fitted with 6 in. × 6 in. triangular fillets. The exit is through a 2½-inch boot valve at the lower end of the tank.

It will be convenient at this stage to discuss the arrangements for the settling of the spent acid in connection with the super-detoluation scheme (see page 63). The full equipment of the tanks is shown diagrammatically in Fig. 17. Of the two spent acid tanks, the one nearer the acid MNT storage tank is used for receiving the spent acid from the super-detoluators. This is kept always full, and, as each charge is pumped from a super-detoluator, the spent acid and acid-MNT flow over automatically into the second spent acid tank and the acid MNT tank respectively. The spent acid overflow consists of a 6 in. × 3 in. lead pipe burned on to one corner of the vat and running to within about 4 inches of the bottom; the overflow is 19 inches from the top of the tank. The acid-MNT skimming line is a 3-inch lead pipe with branches to each vat, skimming taking place 3 inches above the level of the spent acid overflow. This gives an average depth of 18 inches of nitro-body on the separation tank. The spent acid is pumped away from the second (or storage) tank once per shift of 8 hours.

The weakness of the above arrangement is that the overflow of spent acid and acid-MNT takes place before adequate time has been allowed for proper separation. The running in of each charge disturbs the whole mass, with the result that MNT is carried over into the spent acid tank, while the acid-MNT carries over a small amount of spent acid. The insufficient time for separation also results in the acid-MNT carrying over an appreciable percentage of nitric acid on account of its high solubility, which separates out in the acid-MNT storage tank, resulting in very serious corrosion of the bottom of the tank and of the regulus outlet valves.

The scheme was not sufficiently long in operation for this difficulty to be overcome. The alternatives would be :—

(1) Provision of two extra spent acid tanks and the use of all four tanks as separators, the automatic spent acid overflows from tank to tank being removed. The skimming of the acid-MNT and the pumping away of spent acid would thus take place under control after sufficient undisturbed settling, and not automatically, as under the existing arrangement. Four tanks would accommodate the acid and nitro-body from 2 days' working, and this would permit of 24 hours' settling, which is very desirable.

(2) Replacement of the lead-lined acid-MNT tank by an acid-proof brick-lined tank, or a cast iron tank, if corrosion experiments prove the latter to be suitable. Experience on the continuous process washing plant suggests that either of these materials would withstand corrosion by the acid.

(3) Installation of a pre-settling tank to accommodate one charge from the super-detoluator. After settling for a short time in this tank the contents could then be quickly run into the large spent acid tanks, so that the contents of the latter should not be unduly disturbed. This method has been proved very efficient elsewhere.

The spent acid tanks need sludging out once in three months, during which time about 3 inches of dense sludge accumulate. The method of sludging is as follows. The contents of the tank in question are pumped through the sludge valve into the second tank, a special pipe-line being installed for this purpose, with a branch to each tank, as shown in Fig. 17. After the tank has been emptied, a joint on the pipe-line from the sludge-valve is broken, and the tank washed out, the sludge being broken up by means of a jet of water from a fire hose. In the event of an accumulation of solid DNT, the acid is pumped away, and a hole cut in the side of the tank through the lead lining. The nitro-body is then raked out and despatched to the recovery plant.

Acid-MNT.—An extra lead-lined tank (d, Fig. 16), has been erected at each unit to accommodate the acid-MNT resulting from the super-detoluation process. This tank, which is 7 ft. square × 7 ft. 6 in. deep, is fitted with a 3-inch lead inlet from the spent acid tank skimming line, and with a 2-inch outlet leading to the acid-MNT pump.

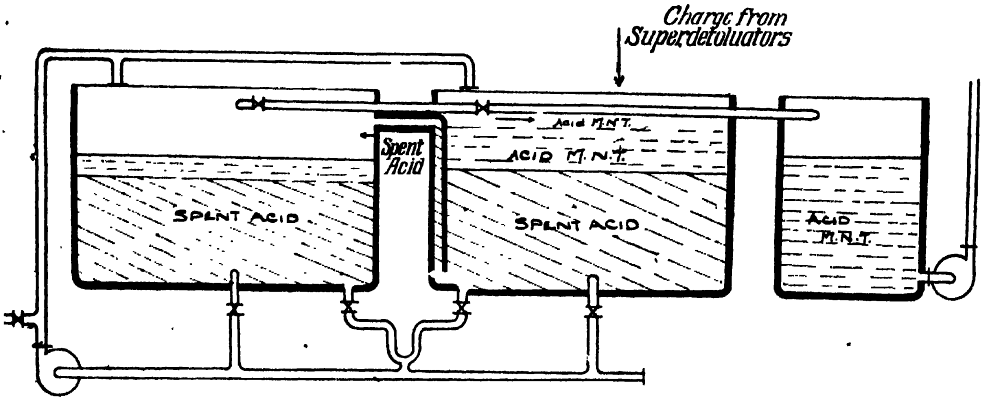

FIG. 17.—DIAGRAM OF SUPERDETOLUATION SETTLING AND STORAGE ARRANGEMENTS.

ARRANGEMENTS FOR ELEVATING THE MIXED ACID.

The outlets from the mixed acid tanks lead direct through a 2½-inch line to the four mixed acid eggs (e, Fig. 16), for supplying the four nitrators, the acid charge for each nitration being one egg-full. In the event of this proving too much (as it would do if the mixed acid composition were altered in the direction of increasing the HNO_3 content) the working capacity of the egg may be reduced either by—

(a) reducing the length of the dip-pipe inside the egg; or

(b) reducing the actual capacity of the egg by the introduction of a number of acid-proof bricks.

Plug-cocks are inserted in the pipe-lines from each mixed acid tank and also to the branch lines to each egg. The glands of these cocks need packing about three times weekly, on account of exposure to the weather.

Each egg is 7 ft. 3 in. long × 4 ft. internal diameter, and has a capacity of approximately 610 gallons. It is constructed of ½-inch mild steel plates, and is provided with three steel saddles, the first 1 inch in diameter, for the admission of compressed air; the second, 3½ inches in diameter, for the discharge of acid; and the third, 2½ inches in diameter, for the admission of acid. There is also a 1½-inch drain outlet and a 16 in. × 11 in. manhole. Under normal conditions an egg fills in about 10 minutes, and is emptied in about 12 minutes. It is constructed to withstand a pressure of 80 lb. per sq. in The acid (specific gravity 1.845) has to be raised 36 feet, and this can be accomplished by a pressure exceeding 36 lb. per square inch; the normal efficient working pressure is 40-50 lb. per square inch.

The supply of compressed air to each mixed acid egg is controlled from the nitration platform, alongside the corresponding nitrator (see Fig. 22). The air line between the control valve and the egg is provided with two exhausts for releasing the pressure, one by the nitrator and one near the egg, the second acting as a safeguard against blowing up a charge of mixed acid into a nitrator already full.

NITRATORS AND SUBSIDIARY PLANT DETAILS.

The general arrangement of the nitrator and its feed tanks, pipe and valve fittings, etc., is shown in Fig. 18, and further details of the nitrator and stirrer in Fig. 19.

Nitrator.—The nitrators, detoluators, and super-detoluators are identical in all respects Each consists of a cast iron vessel, 1¼ in. thick, 6 ft. 7 in. in diameter, and 6 ft. deep, with a rounded base. The cast iron has withstood the conditions very satisfactorily, corrosion being only slight in any case, and then mainly at the level of the acid prior to diluting the charge after nitration is complete. The greatest number of charges worked in any one set is 3,195, corresponding approximately to 3,200 tons of TNT.

The nitrator is cast in two parts, a base and a cylinder, the two being bolted together by means of a faced flanged joint. The joint was made originally with 1-inch blue asbestos tape which had been soaked in silicate of soda solution. A joint made in this manner lasted on an average for 12 months. The experiment was then tried of replacing the asbestos with strip lead. This lasted slightly longer than the asbestos, but ultimately needed replacement, on account of the lead "creeping."

The nitrator covers have not required renewal, but after three years' wear show signs of corrosion at the entry of coils, due to condensation of weak nitric acid at this point, and round the water funnel, due to the water splashing and drips. A rectangular hole in the cover, flashed round with thin sheet lead and closed by a light sheet iron lid, serves for sampling.

Stirring Gear.—Stirring is effected by means of a screw propeller, 20 inches in diameter and with a pitch of 24 inches, which rotates at 160 r.p.m. inside a vertical cast iron cylinder, 22 in. inside diameter × 22 in. deep (Fig. 19). The latter is supported on three mild steel legs, so that the bottom of the cylinder is 8 inches above the bottom of the nitrating vessel and one inch above the level of the cast iron grid which supports the coils

The propeller is built up of cast iron boss with mild steel blades, and is fixed to the end of a 2½-inch vertical shaft belt-driven from a counter-shaft, which in turn is driven from a main shaft running the full length of the nitration house. The propeller lifts the nitro-body and acid from the propeller chamber, and in order to break up the core of the liquid, a disrupter blade, consisting of a piece of twisted 3 in. x ⅜ in. flat wrought iron bar clipped to the shaft, was originally fitted to the shaft just above the top of the chamber. This was found to give unsatisfactory circulation on account of the completeness of the interference of propeller and disrupter. The latter has since been altered so that its action is supplementary to that of the propeller.

Efficient stirring is very necessary, on account of the considerable resistance to circulation caused by the coils and the grid on which they stand. There is a danger of the circulation being effective only in the centre of the vessel, the outer ring of liquid being left almost undisturbed. This actually happened when the propeller and disrupter worked counter to each other.

Outlet and Three-Way Cock.—The outlet from the nitrator is 3½ inches in diameter, and is tapered to form a seating for a regulus metal plug valve. A 3½-inch diameter cast iron bend connects the outlet to a 3-way steam-jacketed cast iron glandless plug-cock (Figs. 20 and 21). The case of the latter is cast with suitable guards, so that the operating lever, which is interconnected with the regulus metal plug valve inside the nitrator, cannot be removed from the slot in the plug of the 3 way cock without bringing the regulus metal plug back into its seat.

Considerable trouble was at first experienced with the 3-way cocks due to:—

(1) *Seizing of the plug in its seating.*—This occurred when steam had been applied to the jacket. Experiment proved heating to be unnecessary and the steam has since been cut off.

(2) *Corrosion of the large face of the plug.*—This difficulty was overcome by the introduction of graphite in the lubricant, which reduced the rate of corrosion, and when this did occur made the seat sound by filling up the corroded parts.

Originally cylinder oil (A.A. Oil Co.'s 600W) was used as a lubricant for the 3-way cock. This, however, was found to lack body and was discontinued in favour of Extra Hecla Cylinder Oil (A.A. Oil Co.'s), which was more viscous, but which proved unsuitable on account of the fact that it contained nitratable oils, to the presence of which were attributed two fires which occurred in the drip-trays below the cocks. After much experimental work a satisfactory lubricant was arrived at, which increased the life of the 3-way cocks from 200 to 1,200 charges. The mixture consists of 45 parts of Dixon's No. 2 Flake Graphite with 55 parts of mineral jelly. Whereas this composition was suitable for use in summer, it was modified to 60 parts of mineral jelly with 40 parts of graphite in winter, the former mixture being too viscous for easy application during cold weather.

Worn out 3-way cocks proved capable of repair by re-boring the bodies and fitting new plugs.

Fig. 21

To face p. 51

Nitrator Run-Off:—The two outlets from the 3-way cock are connected to the separator and drowning pit by means of 3-inch cast iron and 3-inch lead pipes respectively (Fig. 21). Each of these pipes is fitted with a ½-inch air-pipe with 3/16-inch nozzle. The air connection to the drowning-line is used after each separation to make certain that the line is clear before commencing the next nitration. The connection to the run-off pipe to the separator is kept slightly open during the whole time the contents of the nitrator are being discharged. This connection was installed to meet the difficulty which arose whenever a nitration charge needed to be "reinforced" with a further quantity of mixed acid. Reinforcement necessitated running off a part of the acid to the separator. When sufficient acid had been run off and the 3-way cock closed again, an oscillation of pressure took place in the run-off pipe, the positive pressure of which tended to throw the plug of the cock out of its seating, with a danger to the person conducting the operation. The supply of compressed air nullified this effect and entirely removed the danger. Once installed, it was necessary to keep the compressed air valve slightly open during all discharges of the nitrator in order to prevent blockage of the nozzle with TNT.

DNT splash tanks.—The DNT is elevated from the cast iron egg below the detoluator into a cylindrical splash tank from which it flows into the measuring tank feeding the nitrator (Figs. 14 and 18). Each splash tank is 2 ft. in diameter × 2 ft. 6 in. mean depth, with a fall at the bottom of one inch towards the outlet, and is constructed of 3/16-inch mild steel plates with welded joints. The outlet from the tank leads into the DNT measuring tank by a bond without valves. A steel cover is fitted to each splash tank with a centre opening 3 inches in diameter for the discharge of air leading to a common air exhaust main running the length of the building and discharging into the atmosphere.

DNT measuring tanks.—The DNT measuring tanks (marked "MNT measuring tanks" in Fig. 18) supplying the nitrators consisted originally of mild steel cylindrical vessels 3 ft. 6 in. diameter × 3 ft. 3 in. mean depth, having a slope of 2 inches towards the 1½-inch outlet, which was controlled by a plug cock fixed to a boss welded on to the tank. Gauge glasses were fitted to each tank but proved of little value owing to the solidification of the DNT in the glass; hence they were cut out. On the introduction of the higher gravity DNT from the detoluators the tanks were provided with 1-inch steam coils to prevent the DNT from setting. The life of the tanks was about 10 months, and the bottom corroded quickly, this being accelerated by the accumulation of sludge. Replacements consisted of lead tanks, 4 ft. diameter × 3 ft. 6 in. deep, supported by 3 wrought iron bands; these have given entire satisfaction.

DNT funnel.—The DNT funnel (Fig. 18), which was originally made in lead, is 8 inches in diameter tapering to 1½ inches, and is fixed to a lead pipe 18 inches long which passes below the level of the mixed acid charge in the nitrator. The lead funnel became misshapen and collapsed at the burnt joints, and was replaced by a similar funnel in regulus metal.

Water measuring tank.—The water measuring tank (Fig. 18) is a rectangular mild steel vessel, 3 ft. × 2 ft. 6 in., provided with a 1½-inch diameter outlet controlled by a plug-cock on a delivery pipe. As the gauge glass was of little service owing to the height of the tank above the operating platform, an adjustable automatic overflow was provided. This consisted of a vertical swivel attached to the inside of the tank at the existing overflow point, and provided with a 1½-inch wrought iron pipe 12 inches long, screwed into an elbow, which could be raised or lowered according to the volume of water required. The overflow pipe was disjointed near the nitrator, and a funnel fitted to receive the overflow, so that when the required volume of water was in the tank the overflow was always within easy view of the operator, and the water supply could be turned off. This arrangement is shown in Fig. 22, together with the general arrangement of the nitrator and its feed tanks.

Water Funnel.—The water funnel is of lead, fixed to a 1½-inch lead pipe which is extended 20 inches above and below the lid. The life of these funnels is short (14 days), due to :—

(1) Corrosion near the lid caused by condensation of dilute nitric acid on the underside of the lid and the wash up of weak diluted acid, the latter effect being more marked when nitration charges were increased, and the nitrators were used to the limits of their capacity.

(2) Corrosion of the inner walls of the stem at the level of the liquid inside the pan. This is to be accounted for by the water drips from the measuring tank, which diluted the undisturbed mixed acid in the stem. In order to overcome this, the water pipe was fitted to a swivel, so that it could be swung clear of the nitrator, with the result that the life of the funnels was increased to about two months.

A less corrosive metal could not be used for the water funnels as it was necessary to curve their stems according to the position of the coils inside the nitrator.

Cooling and heating coils.—Each nitrator is provided with four concentric coils, each consisting of 11 turns of 1½-inch lead pipe wound at a pitch of 4 inches. The details of the separate coils are given in the following table :—

Coil No.	Diameter ft.	Length of pipe. ft.	Volume. cu. ft.	Internal surface. sq. ft.	No. of supports.
1	6	208	4.55	82	12
2	5	173	3.80	68	10
3	4	139	3.04	55	8
4	3	104	2.27	41	6

Total cooling surface = 246 sq. ft.
Gross capacity of nitrator = 184.5 cu. ft.
Volume of coils and supports = 13.3 cu. ft.
Net capacity of nitrator = 171.2 cu. ft.

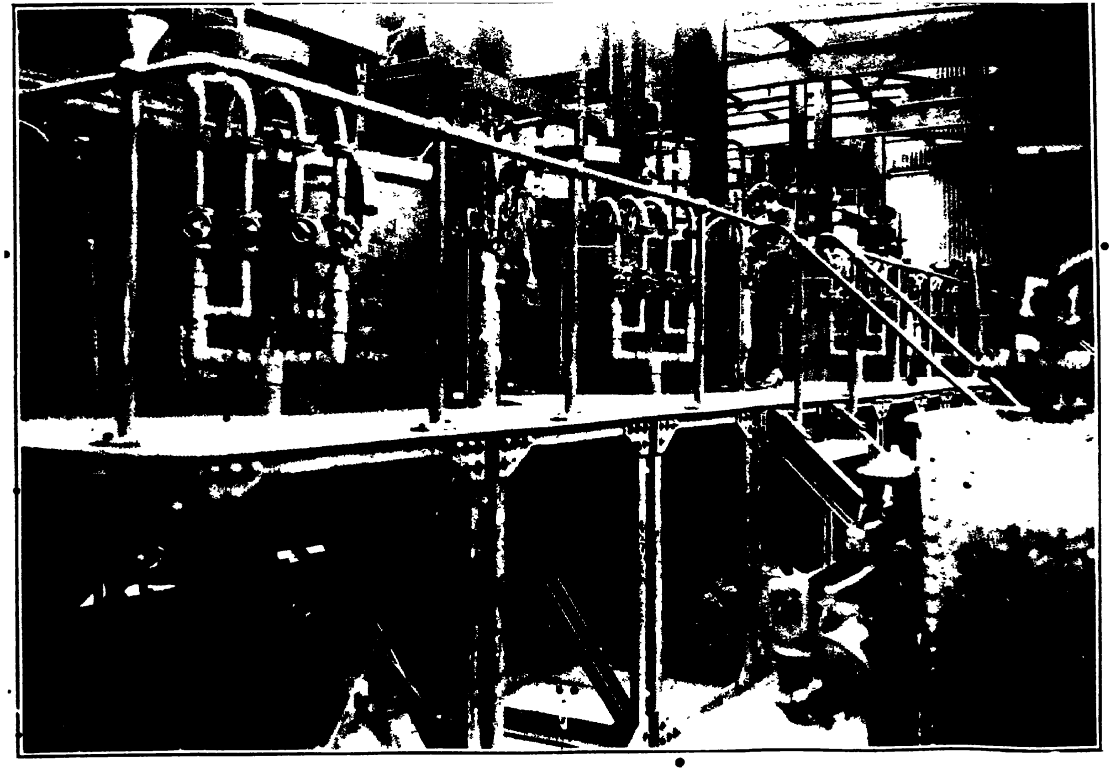

FIG. 22.

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The three largest coils are used as water coils for cooling purposes, and the small coil mainly as a steam heating coil. The latter is connected to the water supply, however, and may be used as a cooling coil in an emergency. Usually one coil (No. 3) suffices for controlling the temperature during nitration. The coil supports were originally made in lead, burned on to the piping, the coils being supported on a cast iron grid constructed in four sections (Fig. 23). The cast lead, though satisfactory in the case of detoluator coils, did not withstand satisfactorily the high temperature in the nitrators. The coils at present are supported by means of cast iron standards which rest on the grid previously referred to. Each coil support, the details of which are shown in Fig. 23, is in two halves which are bolted together with wrought iron bolts, lead bushes being inserted at the points where the lead pipe passes through the supports. The supports are kept in position by 2 wrought iron bands, bolted to each support, thus giving rigidity to the finished coil. The cast iron standards have given complete satisfaction in the nitrators. There have been no signs of collapse, and the only signs of corrosion have been on the bands and standards (on coils Nos. 2 and 3) in the proximity of the bottom of the water dilution pipe, and these were renewed whenever the coils were replaced.

A new set of coils is installed when two of the set are out of commission. The remaining coils, if otherwise sound are put to further use in a detoluator.

Water connections to coils.—The water mains feeding nitrators and detoluators are 6 inches in diameter, and from these mains 4-inch branches are taken off at each nitrator and detoluator. These branches rise vertically to a point just below the operating platforms, where a control valve is situated. The distributing header is connected at this point, and supplies 1½-inch branches which are coupled to the inlets of the four coils, each inlet being controlled by a valve. The 1½-inch branch to No. 4 coil is fitted with a 1-inch steam connection between the coil and the plug-cock. A ½-inch compressed air line is also connected to the header, between the 4-inch valve and the 1½-inch control cocks, this being used only for testing the coils (Fig. 24). The coil outlets (shown in Fig. 22) are similar in size to the inlets, and

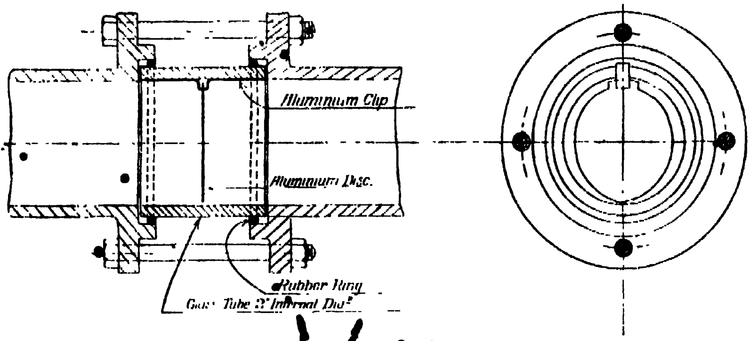

FIG. 25.—DEVICE FOR INDICATING FLOW OF WATER THROUGH COOLING COILS OF TNT NITRATORS.

are coupled together by a similar arrangement, No. 4 coil being fitted with a 1-inch steam exhaust in addition.

The inlet and outlet of each coil is marked with a number in order to avoid confusion, and a safety device similar to that shown in Fig. 25 is inserted between the inlet valve and the coil in the cases of Nos. 1, 2, and 3 coils, to show that water is actually passing through the coil when the valve is opened.

Wear and tear.—Considerable trouble was experienced when the plant was first operated, by the flattening, bursting, corroding, and generally short life of the cooling coils, particularly in the trinitrators. The following were the chief sources of trouble:—

(1) Flattening of the coils due to the high temperature (120° C. maximum) and too great a vacuum (20-25 inches of mercury).

(2) Flattening of the upright portion of the coil inlet, due to the sudden high vacuum produced by closing the plug-cock after the necessary cooling had been effected.

(3) Flattening of the coils at the outlet, due to the high vacuum and temperature previously referred to, together with the unsatisfactory arrangement by which the coil outlets crossed and rested on each other.

(4) Swelling of the bends at the bottom of the inlet lengths of the coils, due to the water-hammer produced on closing the plug-cock.

(5) Swelling and bursting of the steam coils, especially at the bottom rings, owing to leaky steam inlet valves and the coil not being cleared of water before turning on steam.

(6) Corrosion of the inlet and outlet lengths of the coils, due to the water used in washing the top of the lid, and the condensation of dilute nitric acid inside the nitrator, especially on the coil inlets.

(7) Corrosion of the cast iron supports and middle bands of Nos. 2 and 3 coils below the end of the water diluting pipe, due to the marked local dilution.

The following remedies were adopted, whereby the average life of a set of coils was increased from 330 to 1,100 charges:—

(1) The maximum temperature of 120° C. was reduced, at first to 110° C., and later to 100° C.

(2) A release valve was fitted to the outlet water main, which came into operation at a vacuum of 15 inches of mercury.

(3) The inlet arms of the coils were made of much heavier lead (28 lb. per yard).

(4) The water-hammer effect on closing the coil inlet valves was minimised by replacing the plug-cocks by slower opening valves. In addition, 1-inch pet-cocks were introduced on the inlet side of the coils. These cocks were fitted with a plug drilled with a $\frac{1}{8}$-inch hole, the rush of air through which effectually prevented the water hammer. The pet-cocks were shut when the coils were on pressure water (see later).

(5) Steam valves were examined weekly and repaired when necessary.

(6) The crossing over of the coils was avoided by reversing the connections to the outlet header.

Testing of coils.—When new coils are installed, the nitrator is filled with water above the top of the coils and examined for leakages by maintaining an air pressure of 30 lb. per sq. in. in the coils for 10 minutes, the main outlet and inlet valves being kept closed. A ½-inch air connection in the inlet water header is provided for this purpose.

WATER SERVICE IN NITRATION HOUSES.

In order that there shall be no possibility of water entering the nitrating vessel in the event of a coil bursting, the water system is so arranged that the water is sucked through the coils by means of a vacuum pump, instead of being forced through under pressure. The principal water supply for the cooling coils is obtained from the River Dee, and is pumped up into two feed tanks 60 ft. above ground level, each of 300,000 gallons capacity (g, Fig. 13). From these tanks the water flows through ball-valves to break-level tanks (one supplying the nitrators and one the detoluators), in which the water level is maintained a few inches below the uppermost level of the cooling coils which are served by it. The suction end of the pump is connected to the coil outlets, the vacuum being maintained by two Worthington pumps, working alternatively. In the event of both pumps breaking down, or of failure of steam, it is possible to cut out the break level tanks and to run the coils on pressure water from the high level feed tanks at the pumping station. Pressure water is also available from the town main; this, however, is used only in an emergency. The general arrangement of the water service is shown in Fig. 26, and the method of changing over from suction to pressure supply is set out in the water diagram, Fig. 27.

The relative cooling values of river and town water are indicated by the following water temperatures taken in June, 1917:—

4 p.m.	Atmospheric temperature in the sun	28°C.
	Temperature of river water at intake (low tide)	21.5°C.
	" " " reservoir	20.8°C.
	" " " "D" unit	20.5°C.
8 p.m.	" " " flood-tide	21.2°C
	" town supply	12°C.

Location of a burst coil.—The first indications of a leaking coil are obtained at the Worthington pumps, which are provided with small pet-cocks fitted to the upper or discharging compartment, and by means of which the water which has been used for cooling purposes can be tested for acidity. This is done every half hour. The test does not necessarily lead to immediate detection of a leak, as the coils (especially those in the nitrators) are used discontinuously, and it may be some hours before the leaking coil is in use at the time the pump is tested. Leaks in lead coils, however, are insignificant at first and develop slowly, and this method of testing has been found to reveal all leaks before they become serious. When a leak is detected, the location of the coil effected is determined as follows:—

(a) The water in the break-level tanks is tested, as this may be acid due to the acidity of the river water or to leaking super-detoluator coils, the water from which is discharged into these tanks.

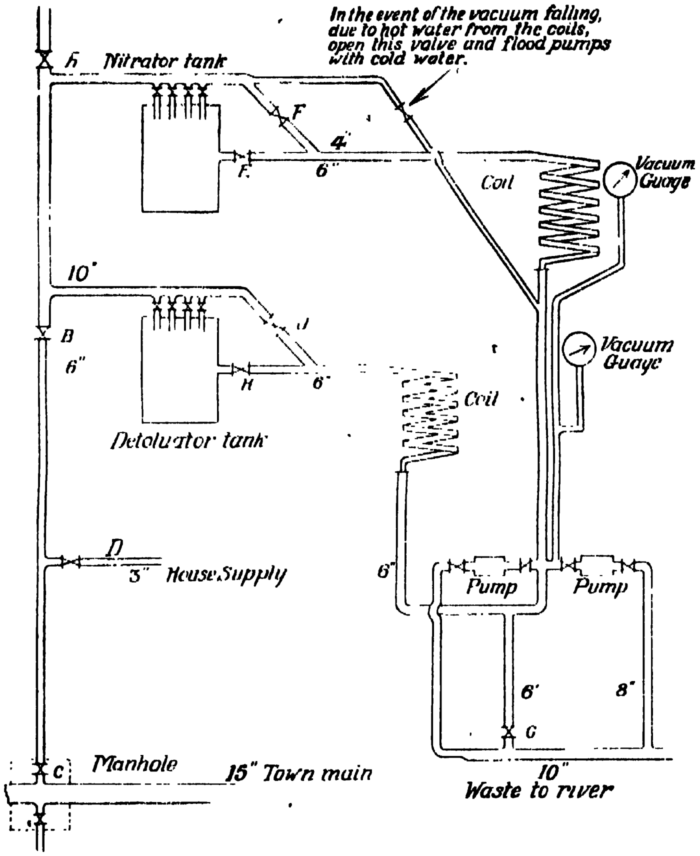

FIG. 27.—WATER DIAGRAM.

A. Reservoir Main Valve.
B. Town Water Valve.
C. Town Water Main Valve.
D. House Supply Valve.
E. Tank to Nitrator Coils.
F. Main to Nitrator Coils.
G. Emergency Outlet from Coils.
H. Tank to Detoluator Coils.
J. Main to Detoluator Coils.

To use emergency supply:—

To Nitrators:—
Close E—open F and G.

To Detoluators:—
Close H—open J and G.

(b) The cooling system is changed over to pressure water supply, and the water from each coil is tested by means of the pet-cock fitted to the outlet header. If the leak is slight, care must be taken to test the first water running from the coil.

FIG. 28.

To face p. 57.]

SEPARATOR AND SUBSIDIARY PLANT DETAILS.

Separator.—The separator consists of a rectangular cast iron vessel 7 ft. 6 in. wide × 4 ft. 3 in. mean depth, the bottom being slightly roundec across the width, and having a slope of 1½ inches in the length towards a 3½-inch diameter outlet. The separator is cast in two pieces, being split by a horizontal flanged joint near the base. This joint was made originally with 1½-inch asbestos tape, which lasted about 18 months; renewals were made with strip lead (see page 49). To the outlet is fitted a cast iron elbow with a regulus metal plug and seating and a 3-way cock, so that the contents of the separator may run either to the sight box or drowning tank. The gear operating the plug-valve and 3-way cock is inter-connected as in the case of the nitrator (see Fig. 28). Two 1-inch steam coils are provided, which rest on a sheet of 12 lb. lead. The separator, which is insulated with a 3-inch coating of asbestos lagging, is covered with an aluminium lid (Fig. 28).

Separator outlet and sight-box.—The three-way cock is connected by a cast iron pipe to a cylindrical sight-box (shown in Fig. 28). This was originally 18 inches in diameter and 30 inches deep, constructed of 20 lb. sheet lead, but this material was later replaced by cast iron, which has proved far more satisfactory. The outlet leads through a 3-way cock alternatively to the once-used acid line feeding the detoluator, or to the TNT line leading into the pelleting vat. Both these lines were at first made of lead, but this material was later replaced respectively by cast iron and silicon-iron, since when no trouble has been experienced.

It may here be remarked that continued experience in the working of the plant, as originally laid down, has resulted in the replacement of lead piping by wrought, cast, or silicon-iron in almost all cases in the nitration house. Not only do these metals provide greater rigidity, but they are capable of easy support, and withstand the necessary wear and tear much more satisfactorily. Ease and quickness in replacement is essential, and the difficulty of lead-burning in an explosive building is thus obviated.

DETOLUATORS AND SUBSIDIARY PLANT DETAILS.

Detoluator.—The detoluator, Fig. 29, is similar in every respect to the nitrator, except that it has no drowning line, and the outlet valve, which was originally a steam-jacketed 3-inch cock, has since been replaced by a 3-inch regulus metal pot-valve. When the higher gravity DNT was introduced there was occasional blockage in the lower part of the pot-valve. This was difficult to clear without removing the plug, which was an exceedingly dangerous practice unless the detoluator was empty, and specially designed 3-inch all cast iron gate-valves (shown in Fig. 29) were introduced satisfactorily as replacements became necessary. Corrosion is more evident in the case of the detoluators, due to the introduction of weak nitric acid with inefficient stirring. This results in the eating away of the vertical stirring shaft at the acid level. The coils in the detoluator last somewhat longer than in the nitrators on account of the lower reaction temperature, the life in this case being about 1,250 charges.

MNT measuring tanks.—The original tanks rested on weighbridges, which necessitated the run-off being disjointed from the pipe delivering

MNT to the detoluator. The weighbridges were afterwards removed, being far from satisfactory, and the tanks were replaced by somewhat larger cylindrical mild-steel tanks, 3 ft. 6 in. diameter × 3 ft. 3 in. mean depth, with a capacity of about 2,300 lb. of acid-MNT of 1.20 specific gravity. No trouble due to corrosion was actually experienced in the case of these tanks, but corrosion would almost certainly have arisen in time owing to the acidity of the acid-MNT. Such corrosion could be minimised by increasing the spent acid storage capacity, as suggested on page 47. In the event of this measure proving inadequate, lead tanks would be essential. Approximately one inch of sludge accumulates in these tanks per month. This sludge is gritty, and passes into the 1½-inch outlet, damaging the face of the regulus screw-down valve situated above the detoluator platform. This trouble has been overcome, however, by fitting the valves with renewable rubber seats.

Water dilution tanks.—These are similar to those supplying water to the nitrators.

Nitric acid pipes.—The pipes supplying weak nitric acid from the receivers at the absorption towers to the detoluators are of two types, silicon-iron and earthenware, both 2 inches in diameter, and controlled by two 2-inch silicon-iron cocks or earthenware block-valves at the receiver and detoluator. The pipe is run directly into the detoluator, dipping one inch below the lid. A sight glass is inserted in each branch-line between the cock and the detoluating vessel. The earthenware pipes proved the more satisfactory, the silicon-iron pipes proving somewhat porous. The latter were consequently treated with silicate of soda before being erected, the joints being made with an asbestos ring soaked with silicate of soda.

Sight-boxes.—The sight-boxes for the detoluators (Fig. 29) are of lead, similar to those originally installed at the separators; these have not needed replacement owing to the much lower temperature to which they are subjected. The sight-box has a 3-inch exit for spent acid and a 2-inch side exit for DNT.

Spent acid pipes.—The spent acid pipes from the sight-boxes are 3 inches in diameter, leading to a common 3-inch main to the spent acid tanks, with branches to the super-detoluators. All these pipes are supported on channel irons. The only trouble experienced has been due to blockage on account of the accumulation of DNT in the pipe, this being due to the lack of fall on the spent acid pipe. For this reason, the pipes are flanged in lengths not exceeding 9 ft., with a branch for steaming purposes. The branches to the two super-detoluators are controlled by 3-inch all cast iron gate-valves.

DNT egg.—After running off the spent acid from the detoluator to the super-detoluator, the DNT is run down to a cast iron egg, 2 ft. 8 in. diameter × 6 ft. 7 in. long, with a manhole and cover at each end, see general arrangement Fig. 14. The capacity of the egg is 191 gallons. Four 2-inch diameter openings are cast in the body of the egg, one for admitting the DNT, one for the dip-pipe, one for the compressed air inlet, and the fourth

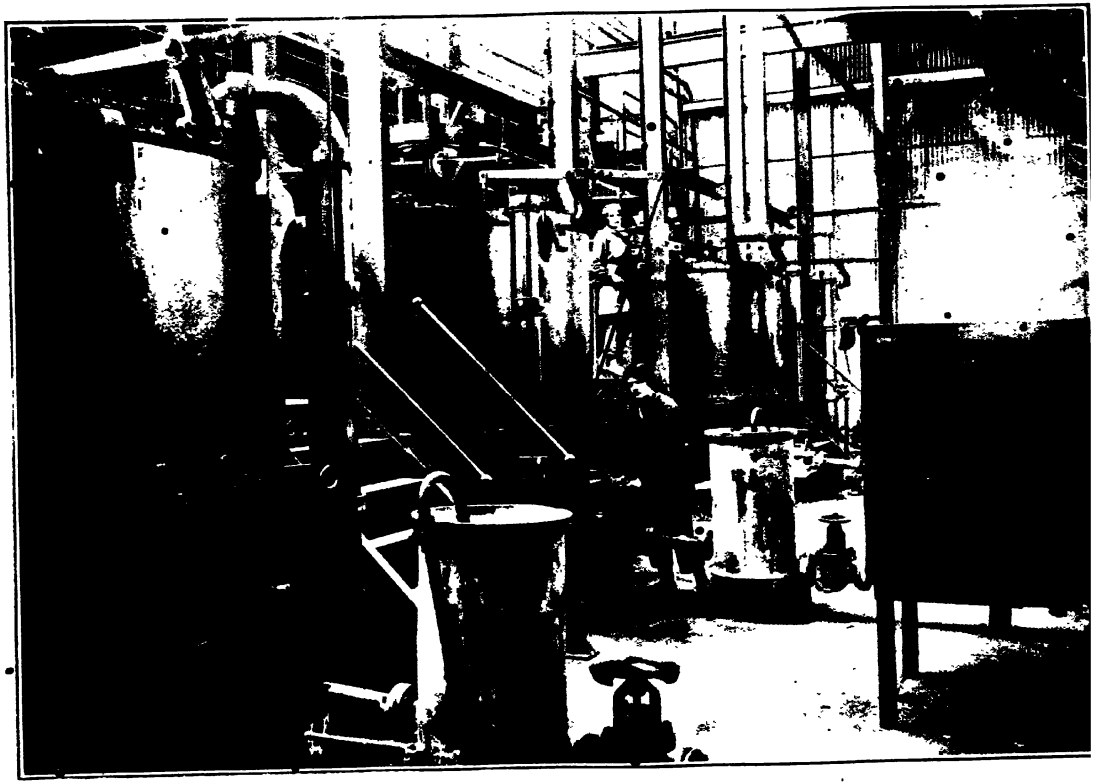

FIG. 20.

To face p. 55.

(a spare) being blanked off. The metal is 1¼ inches thick, all joints being machine faced and made with asbestos soaked in silicate of soda.

At one time, a certain amount of trouble was experienced, due to the setting of the high gravity DNT in the eggs, particularly in cold weather. It was first sought to overcome this by enclosing the egg in a 4½-inch brick wall, the tank so formed being filled with water which was heated by an open ½-inch steam-pipe. The walls collapsed, however, and the next attempt consisted in totally enclosing the egg by brick walls and a tiled cover, the enclosed space being kept warm by means of a steam radiator. This was quite successful, no appreciable fall in temperature taking place, even when nitro-body remained in the egg for several hours.

DNT pipes.—The DNT delivery pipe from the DNT egg to the nitrator splash-tank was originally a 1½-inch unjacketed wrought iron pipe. The diameter of this pipe was afterwards increased to 2 inches. This delivery line was jacketed by a 4-inch pipe, and heated by a ½-inch wrought iron steam-pipe which ran between the delivery line and the jacket. The heating was necessitated by the higher setting point of the DNT.

SUPER-DETOLUATORS.

These vessels are similar to the detoluators except that only 3 cooling coils are provided, the large coil being omitted. Cooling water is supplied under pressure from the nitrator supply-tank, and returns to the detoluator supply tank.

After super-detoluation, the whole charge of spent acid and nitro-body is pumped from the vessel to the spent acid tanks by means of a direct driven 10¼-inch impeller centrifugal pump. The general arrangement is shown in Fig. 14.

PRELIMINARY WASHING PLANT.

The TNT from the separator sight-box at one time passed directly into the pelleting vat. In the nitration houses provided with preliminary washing plant, however, the TNT is given a preliminary wash with hot water before pelleting. Each preliminary washing plant comprises a washer, sight-box, and hot water tank.

Preliminary washer.—This consists of a cylindrical silicon-iron vessel, 4 ft. diameter × 5 ft. deep, having a rounded base. The vessel is cast in two pieces, with a flanged joint which is made with a strip of lead and acid-resisting cement. The plant was not in operation long enough to determine the effective life of the silicon-iron material used, or its suitability for the purposes of preliminary washing. There were certainly signs of serious corrosion, and it was intended, had the pots needed early renewal, to replace them with wooden vats or cast iron tanks lined with acid-proof bricks, similar to the TNT storage tank at the continuous washing plant (see page 81). Each washer is fitted with two 2-inch inlets, 11 inches from the top, for the introduction of TNT from the two separator sight-boxes feeding the set; two 2-inch outlets, 22 inches from the top, one for each detoluator fed by the set; and one 2-inch outlet at the bottom, for the washed TNT passing through the sight box to the pelleting tank. All connections and cocks are of silicon-iron. An agitator is provided, 24 inches in diameter, the later type of which is made in silicon-iron and has a disrupter blade of 36 inches pitch. A steam-pipe of 2-inch silicon-iron, perforated at its lower end with two ¼-inch holes, is provided for heating the charge.

Sight-box.—This is rectangular in shape and also of silicon-iron, 15 in. × 9 in. × 16 in. deep, with an end inlet and a bottom outlet, through which the TNT flows to the pelleting tank by means of a 2 inch silicon-iron pipe (Fig. 14).

Hot water tank.—A 50 gallon galvanised iron tank is provided to supply hot water to the washer. This is provided with water and steam inlets, and with a water outlet.

PELLETING VATS.

Construction of vats.—Each nitration set is provided with a pelleting vat constructed of pitch pine of inside dimensions 9 ft. × 5 ft. × 3 ft. deep. The method of draining the water from the vats is by means of a wood draining box fitted to the bottom of the vat. This consists of a rectangular box, 4 ft. × 1 ft. 6 in. × 1 ft. 6 in. deep, which is filled with quartz (Fig. 30). Through one end of this, at about 6 inches from the bottom, is fitted a 3-inch perforated lead pipe reaching the length of the box. On the outside of the box this pipe is reduced in diameter to 2 inches and is fitted with an earthenware plug-cock, this being operated to drain the tank. Between the earthenware cock and the end of the drain box, a 1 inch compressed air connection was first of all provided, the intention being to blow back the small particles of TNT which tend to choke the perforations in the lead pipe. The air connection was afterwards changed to a 1½-inch water pipe connected to the general water service, as it was found to be much more effective to wash out the small TNT particles than to attempt to blow the perforations clear; this wash-back connection is utilised for filling the vat with water. The TNT is pelleted by means of a 1-inch swivel jet, connected to the water supply (Fig. 30).

Method of emptying pelleting vats.—The vats were originally so designed that it was necessary to dig out the pelleted TNT ("pellite") with wooden shovels, and discharge it through a plug hole, 12 inches in diameter, in the bottom of the vat, the TNT falling through a chute into tip-wagons on the basement floor below. The task of discharging the pellite was much facilitated by providing a door in one end of the vat, and drawing the TNT over the plug hole by means of an iron rake inserted through the opening (Fig. 31).

In the design and construction of the latest pelleting vats full advantage has been taken of the experience gained in operating the older types. The principal advantages of the new type (Fig. 30) are as follows :—

(a) Efficient drainage by means of the drain box, and method of filling the tank with water through the wash-back connection.

(b) Ease in discharging the TNT from vat to tip-wagon is secured by building the vat with an inclined bottom, and raking the pellite

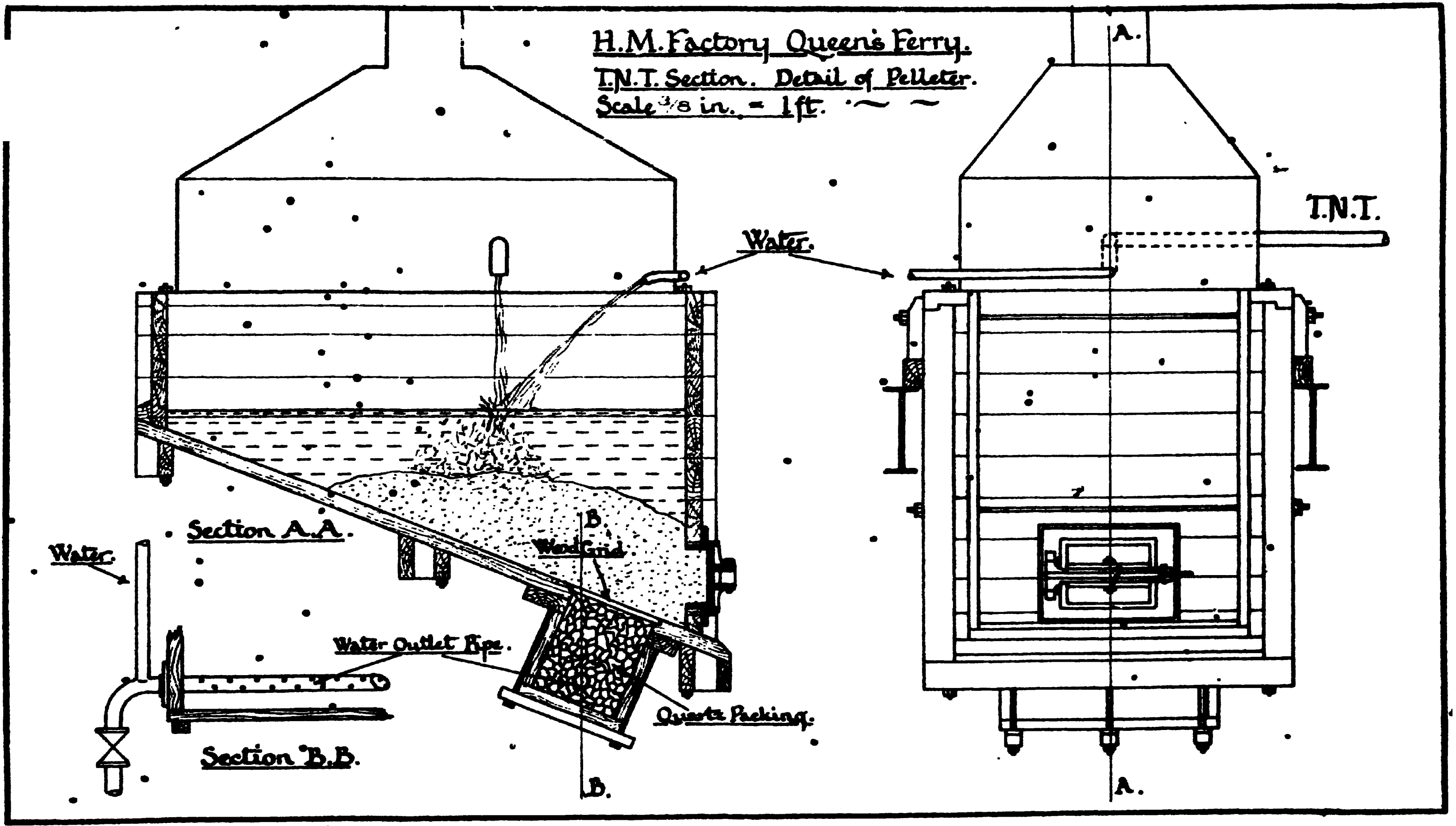

Fig. 30.

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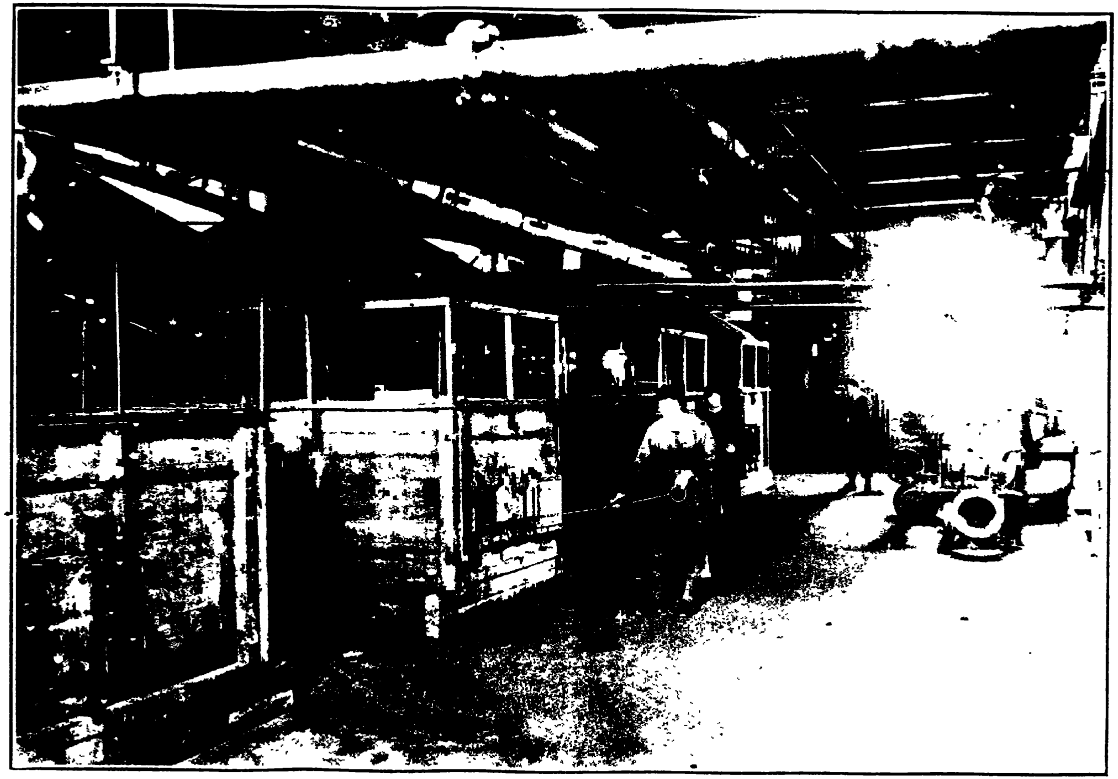

FIG. 31

To face p. 52.

direct into the wagon through the rectangular opening in the end of the vat. The opening is surrounded by a cast-iron frame, which carries a door of the same material. The door is closed and made water-tight by means of a single wing-nut and screw; the weight of the door is taken by the hinge between the door and the frame.

Draught arrangements.—Each pelleting vat is provided with a light heet iron hood, which communicates by means of a damper with a 24-inch luct, through which a suction is induced by a pottery ejector. The latter s supplied with air by a 6-inch branch from the fan used in connection with he absorption towers. The fumes from the pelleting process, which conain very little nitrous fumes, are discharged direct into the atmosphere.

Operated on the above lines, the pelleting process, which gives rise to extremely objectionable fumes, is carried out with the minimum of inconvenience to operatives, and it will be seen from Fig. 31, which shows the method of emptying the earlier type of vat,[*] that the operation is conducted with absolute freedom from fumes.

DROWNING TANK.

In view of the possibility of the temperature of a charge in a nitrator getting out of control, and the desirability of running the charge into water, a drowning tank is installed at each unit. This tank is 25 ft. × 20 ft. × 7 ft. 6 in. deep, with 18-inch brick walls increasing to 27-inch at the base, built in cement except for the inner course which is built in pitch. This was unsatisfactory, and in one or two units in which the drowning tanks have leaked badly, the joints have been raked out and re-pointed with acid-proof cement, which has held satisfactorily.

PUMP HOUSE.

The pump house adjoining the nitration house contains 2 centrifugal pumps with 10¼ inch impellers, which are direct-coupled to enclosed motors. One of these pumps delivers spent acid to the denitration plant at the Acids Section, and the other performs two duties, viz., elevates MNT from the MNT storage tank to the super-detoluator measuring tanks, and the acid MNT from its storage tank to the detoluator feed tanks. These pumps have been operated very successfully, and have proved particularly suitable for the duties required. The only trouble experienced with them is due to leakage at the gland, which requires re-packing every 5 days in the case of the MNT pump, and every 10 days in the case of the spent acid pump, *i.e.*, after discharging upwards of 1,000 tons of acid. The packing material found most suitable consists of ½-inch blue asbestos cord, boiled for 24 hours in a mixture of equal parts of tallow and mineral jelly. The working pressure of the pumps varies from 20-35 lb. per sq. in., according to the distance over which the acid is pumped. In the event of the spent acid pump failing, a lead-lined egg of 6 tons capacity is provided for blowing the spent acid to the denitrators by compressed air. These eggs have been used about twelve times in all, and their use has been rendered quite unnecessary since the installation of centrifugal pumps.

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[*] This photograph, which was taken in a nitration house closed for repairs, shows the detachable side-pieces of the fume hood removed. The Worthington pumps for circulating water through the cooling coils are seen on the right.

Spent acid line.—A 2-inch lead pipe-line, for the delivery of spent acid, runs from each nitration house to the Acids Section, the five separate pipes being connected at the denitration plant to two 5-inch headers (one acting as spare) which deliver to the storage tanks. The only repairs which have been necessary to the spent acid pipes have been of a minor character, due to leakage at the joints.

A lead pipe is also provided for returning liquid nitro-body from the denitrators to the spent acid tanks at "A" and "B" units. This has needed repair continually, due to the corrosion of the line by weak nitric acid which has not been completely separated from the nitro-body at the denitration plant. The main part of the nitro-body consists of MNT condensed in the Hart condensers at the denitration plant. This is passed, together with the condensed nitric acid, through a system of separating bottles in which the nitric acid is diluted in order to bring it below the gravity of the MNT and thus permit separation of the liquids.*

ABSORPTION OF FUMES EVOLVED FROM NITRATORS, SEPARATORS, AND DETOLUATORS.

Pottery fume mains, to which each nitrator, separator and detoluator is connected by 4-inch pottery pipes, pass beneath the floors of the nitration house. The three fume mains are brought together in a 15-inch main outside the nitration house, and the fumes are drawn through a set of 8 absorption towers (f, Fig. 16) by suction induced by a 20-inch fan. Each tower is 36 inches in diameter and is loosely packed at the base with acid-resisting bricks, plenty of free space being left at the inlet to the tower. The chequer brick-work serves to support a further quantity of packing, which consists of 4,000 closely packed "rings," the latter reaching to within 12 inches of the distributor at the top of the tower. Water is fed in at the top of the last tower, and is circulated throughout the system by means of Pohle air lifts. The weak nitric acid (50-55 per cent. HNO_3) which flows from the first tower is elevated to one of two rectangular receivers, 4 ft. × 3 ft. × 3 ft. deep, in which it is allowed to accumulate over a period of 12 hours. During this time the contents of the second receiver are run at intervals into a circular receiver, 36 inches in diameter, from which the acid is run to the detoluators as required. Since the installation of these towers, recoveries have been determined with great care and accuracy over long periods, and it has been established that a recovery of 102.5 lb. of 100 per cent. nitric acid per ton of TNT produced is obtained.†

SHAFTING AND BELTING.

The stirring gear in the nitrators, detoluators, and super-detoluators was originally operated from 3 lines of shafting, belt driven from motors. The

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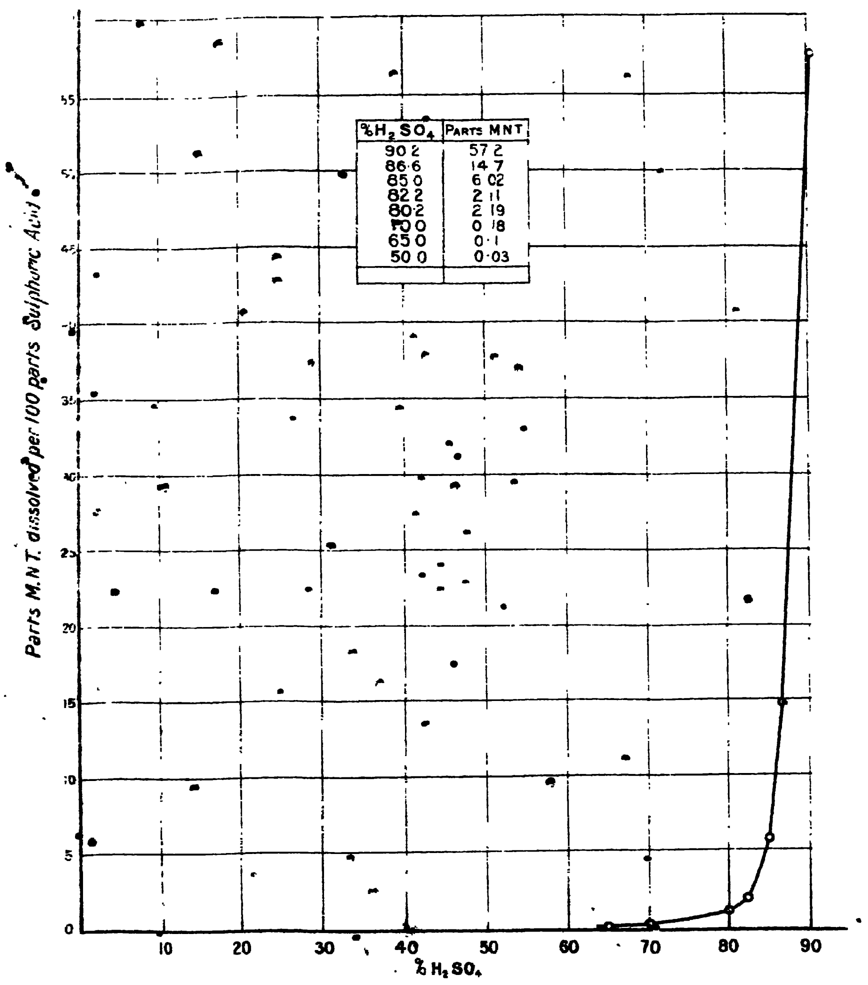
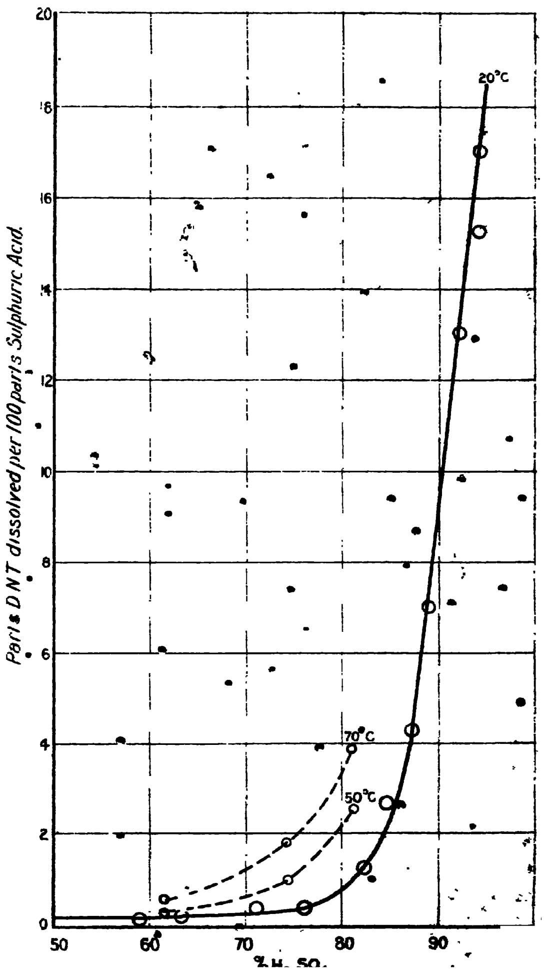
* Details of the recovery of nitro-body at the denitrators are given in the first publication of this series.

† For details of the absorption towers see first publication of this series.



%H_2SO_4	Parts MNT
90·2	57·2
86·6	14·7
85·0	6·02
82·2	2·11
80·2	2·19
70·0	0·18
65·0	0·1
50·0	0·03



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shafting carried friction clutches which transmitted the drive to the corresponding stirring gear by means of a belt given a ¼-twist to the vertical agitator shaft (Fig. 18). No change has been made in this arrangement as regards the detoluators, although the stirring was never thoroughly satisfactory on account of the failure of the wood faces of the friction jaws due to the acid atmosphere. In the case of the super-detoluators the friction clutches were removed and fast pulleys fixed in their place, as it is not necessary to stop stirring to allow separation to take place, the whole charge being pumped to the spent acid storage tank (see later). The whole system of driving was altered, however, in the case of the nitrators. A new line shaft was fitted along the back of the building, and the original was then cut up to make four counter shafts, each of which carried a fast and loose pulley, and a fast pulley to drive the agitator. Five-ply Balata belting is used throughout, the motor to line shaft belt being 5 inches wide, line shaft to counter shafts 3 inches, and counter-shafts to nitrators 4 inches.

Two 12 H.P. motors are used to drive the nitrator and detoluator stirrers, and a 5 H.P. motor to drive the super-detoluators.

COLOURING OF PIPE LINES.

All pipe lines throughout the plant are painted with distinctive colours according to a uniform scheme, as a result of which any perplexity and confusion are avoided.

In the description of the trinitration process which follows, the various stages will be considered following the flow of nitro-body during its gradual nitration from MNT to TNT.

SUPER-DETOLUATION.

General.—A super-detoluation scheme is only necessary when the nitration of toluene is carried out in two distinct operations, as is the case at Queen's Ferry. If, however, the spent acid from the detoluators is further utilised for mononitration (revivification being necessary in this case), it is super-detoluated automatically during the mononitration process.

Spent acid after detoluation contains as much as 1-2 per cent. of nitro-body, which consists almost entirely of DNT and MNT, each of which is soluble in spent acid containing 70 per cent. of H_2SO_4 to the extent of about 0.3 per cent. (see solubility charts, Fig. 32). In addition to nitro-body in solution, however, a certain amount of nitro-body is held mechanically by the acid for a considerable time. Prior to the introduction of the super-detoluation process, the bulk of this settled out in the spent acid storage tanks, but a considerable quantity was always carried to the denitration plant on account of the insufficient time allowed for settling, in view of the lack of storage accommodation. Some of this, chiefly DNT, settled out in the storage tanks at the denitrators and was returned to the TNT plant. The MNT was recovered during denitration, being steam distilled and condensed in Hart condensers. The DNT remaining in the denitrated acid distilled over at the higher temperature obtained in the process of concentration, sufficient of it being thrown down again in the Cottrell precipitator at the Gaillard towers to cause serious blockage troubles. The greater part of the DNT, however, escaped up the precipitator stack and was lost.

The DNT is removed by washing the spent acid with MNT before the latter is charged to the detoluators, leaving theoretically about 0.3 per cent. of MNT as the total nitro-body in solution. In practice, the actual figure is between 0.4 and 0.5 per cent., owing to incomplete separation, and in any case this is all recoverable. The actual saving in nitro-body by this scheme amounts to approximately 2 per cent. of the total consumption of MNT, equivalent to a reduction in the cost of TNT by about £1 4s. per ton. As will be shown later the introduction of super-detoluation also permitted the modification of the detoluation process itself.

Process.—Twice used acid (T.U.A.) is run in from the detoluators at a temperature of about 50° C., and simultaneously is added a measured quantity of MNT, roughly equivalent to the charge of acid-MNT required for the detoluation stage. The balance of the dilution water is then run in, the remainder having been added during the nitration and detoluation stages. All cooling coils are in operation throughout this procedure, and stirring and cooling are continued until the temperature is below 25° C. in summer, and 20° C. in winter. The mixture, the stirrer being still in operation, is then pumped to the spent acid tank which acts as a separator. The whole programme occupies normally about 1½ hours.

Experience has shown the necessity of running the twice used acid and MNT into the vessel simultaneously. When the T.U.A. was introduced before the MNT charge it tended to become "soupy" due to cooling, and the nitro-body was thrown up to the surface out of the solution; it then took a considerable time to dissolve. The addition of the last fraction of the dilution water at this stage is purely a matter of convenience, in order to permit of as large a charge as possible in the detoluator. The actual amount of water added will be given later.

DETOLUATION.

General.—A perfect detoluation process would consist in mixing the MNT charge for the trinitrator with a once used acid containing no nitric acid. This would yield a spent acid containing practically no TNT or DNT, and is essentially what happens in the present super-detoluation process.

Detoluation was fairly satisfactory in the early days, when no nitric acid was added to the detoluators, and when the only nitration of the MNT effected was due to the HNO_3 content (about 2.5 per cent.) in the once used acid at the end of the trinitration. The installation of the present absorption system resulted in the recovery of approximately 100 lb. of HNO_3 (in the form of 50 per cent. acid) per charge and it was decided to add this during detoluation. This scheme yielded a nitro-body ("DNT") having a specific gravity of 1.29-1.30, and consisting of approximately:—

	Per Cent.
TNT	15
DNT	35
MNT	50
	100

Later on, after super-detoluation had been introduced, the efficiency of the detoluation process as such was not so imperative, and it was developed into an almost complete di-nitration stage. This change was necessitated by the acuteness of the weak acid position on the Acids Section, owing to the large amount of 50 per cent. nitric acid which accumulated from various sources in the factory. As it was not expedient to concentrate this acid by means of sulphuric acid, it was decided to introduce it into the detoluators, which formed a convenient outlet for this material. An alternative scheme would be to reinforce TNT *spent acid* with this weak nitric acid, and utilise the resulting mixture for the nitration of toluene to MNT. A slight modification of this procedure was adopted successfully at H.M. Factory, Oldbury, but a full revivification scheme is only practicable where the mononitration plant is conveniently near the nitration plant, and in the present instance the original lay-out and design of plant precluded the possibility of adopting a similar scheme.

The weak nitric acid is transported in 4 earthenware receivers, each 4 ft. × 3 ft. × 3 ft., installed in two railway wagons. The acid is discharged from these receivers into a similar vessel on the ground level, from which it is elevated by a system of 3 Pohle air-lifts to the two main receivers at the absorption towers. Ultimately, in addition to the acid recovered at the nitration house absorption system (*i.e.*, 200 lb. of 50 per cent. nitric acid per nitration), 400 lb. of 50 per cent. acid were taken from other sources in the factory and added to each detoluation charge.

Process.—The once used acid is run down from the separator to the detoluator at a temperature of about 80° C., the stirrer is started, and all the cooling coils turned full on; the charges of acid-MNT, weak nitric acid, and water from the preliminary washer are then added in succession at such a rate as to keep the temperature between 75° and 80° C. After stirring for half an hour at this temperature, the charge is cooled to 50° C. and the dilution water is run in. A further 15 minutes stirring is given to ensure thorough mixing, after which the stirrer is stopped, and thirty minutes are allowed for settling. The specific gravity of the nitro-body is then taken, and the charge separated, the acid being run to the super-detoluator, and the nitro-body to the DNT egg. The whole cycle occupies about 3½ hours.

A facsimile of the detoluation log-sheet is reproduced on the following page.

Control of acid-MNT charge.—The control of the nitration cycle in the above process consists mainly in the adjustment of the acid-MNT charge for the detoluator. As a general rule the whole of the nitro-body from the detoluator is charged into the nitrator; the exceptions are as follows. If the charge has been miscalculated and is too high (as shown by a low gravity in the resulting DNT), the charge to the nitrator is measured as so many sight boxes, and the balance of the nitro-body is run into the super-detoluator or to the spent acid tank. If by any chance such a charge does get as far as the nitrator, there is delay in attaining the setting-point due to insufficient nitric acid, and it is then necessary to reinforce the charge of mixed acid. If the charge is too small, this is revealed by the "dip" of nitro-body in the measuring tank feeding the nitrator. When the original programme was in

DETOLUATION RECORD.

Charges:—

MNT..................lb. (S.G....................)

HNO$_3$..................lb. (S.G....................)

Water..................lb.

The MNT should be run in between.........&........

Unit.... ....

Pan...................... (The Final Trinitration of this N'body is recorded overleaf)

Batch.................. Date ....

O.U.A. Charge started at...............................

Stirrer stopped............................

Separation started..................Finished ....

Total Time............................

Nitric Acid may only be added to a pot which already holds O.U.A., and it must be all in before diluting the charge.

Duration. Hrs.	Duration. Mins.	Time. Hrs.	Time. Mins.	Temperature Observed.	Remarks.
	0				Lubricators given ½-turn by
	10				
	20				
	30				
	40				
	50				
	0				
	10				
	20				
	30				CAUTION.
	40				KEEP
	50				LIDS
	0				ON
	10				FUNNELS.
	20				
	30				
	40				
	50				
	0				Final Product
	10				S.G.
	20				
	30				DNT Tank dips.
	40				Before blowing inches
	50				After blowing inches
	0				Difference inches
	10				
	20				DNT separated and blown by
	30				

Signatures
Operative commencing...
Handed over to..

operation, working with a low gravity DNT, a small charge was also revealed by an abnormally high gravity of the DNT. The "dip" of the nitro body was at once taken, and acid-MNT run in to make up the normal charge.

On the final programme, however, in the event of a small charge, the gravity did not rise above 1.37 (the normal gravity being 1.35), and the result of the smallness of the charge was simply that nitric acid was left in the twice used acid.

The actual amount of the acid-MNT charge for any detoluation obviously depends (a) upon the specific gravity of the acid-MNT, which, with normal running, is fairly constant at 1.19. (b) The HNO_3 content of the once used acid, which varies between 2.0 and 5.0 per cent. This can be allowed for on the basis that 1.0 per cent. extra HNO_3 in the twice used acid corresponds to an extra charge of about 50 lb. of acid-MNT. (c) The amount of HNO_3, as weak nitric acid, available for charging to the detoluator. This quantity is usually constant at 600 lb. of 50 per cent. acid (300 lb. HNO_3), and varies only occasionally due to irregular supplies of acid. An extra 100 lb. of HNO_3 is equivalent to an extra acid-MNT charge of about 50 lb. (d) The amount of nitric acid in the wash-water from the preliminary washer. This is normally about 60 lb., and of necessity must be assumed to be constant. It varies according to the care with which the separation is made at the nitration stage. (e) The HNO_3 content of the mixed acid. This factor is not of great importance as latterly the acid was mixed within 0.1 per cent. limits, and this means only a variation in the charge of 10 lb., which is negligible.

The chief variable factor is the percentage of nitric acid in the once-used acid. At one time it was customary to arrive at this figure by a process of inference from the gravity of the MNT. This meant that there was a lag of two charges between the reading of the gravity and the controlling of the charge. This was very unsatisfactory, as any cumulative variation in the HNO_3 content of the once used acid had time to develop before any attempt could be made to rectify it. Further, a high gravity was often due to bad separation, with the result that, under this system of working, successive charges were varied very widely and the nitrations were by no means uniform. The following table of charges was drawn up for their regulation in the manner suggested:—

Control of MNT charges.—

Percentage of HNO_3 in the once used acid.	No. of inches of nitric acid added from absorption towers and receivers.				
	4 in. lb.	6 in. lb.	8 in. lb.	10 in. lb.	12 in. lb.
1.0 1.1 1.2	1,600	1,625	1,650	1,675	1,700
1.7 1.8 1.9 2.0	1,625	1,650	1,675	1,700	1,725

Percentage of HNO_3 in the once used acid.	No. of inches of nitric acid added from absorption towers and receivers.				
	4 in. lb.	6 in. lb.	8 in. lb.	10 in. lb.	12 in. lb.
2.1 2.2 2.3 2.4	1,650	1,675	1,700	1,725	1,750
2.5 2.6 2.7 2.8	1,675	1,700	1,725	1,750	1,775
2.9 3.0 3.1 3.2	1,700	1,725	1,750	1,775	1,800
3.3 3.4 3.5 3.6	1,725	1,750	1,775	1,800	1,825
3.7 3.8 3.9 4.0	1,750	1,775	1,800	1,825	1,850
4.1 4.2 4.3 4.4	1,775	1,800	1,825	1,850	1,900

It will be seen from the above that an analysis of the HNO_3 content of the once used acid is necessary before the charge can be accurately determined. To arrive at this figure, a sample of once used acid is taken at the commencement of the separation and analysed immediately, the result being available within a quarter of an hour or twenty minutes. This method of control leads to much more uniform operation of the process, and is absolutely necessary when TNT of specially high setting-point is required.

The accompanying table gives a series of fairly consistent results obtained for the setting-points of nitro-body from the detoluators:—

Sp. Gr.	Setting-point.* °C.	Sp. Gr.	Setting-point.* °C.
1.310	24.0	1.360	42.1
1.315	29.2	1.364	44.5
1.320	30.4	1.366	44.0
1.322	33.2	1.370	42.4
1.330	35.0	1.380	40.0
1.340	38.2	1.390	36.0
1.350	35.0		

TRINITRATION.

The aim of the trinitration process is to attain as complete a nitration as possible in the minimum possible time. Complete nitration is mainly a function of composition of the mixed acid, and temperature and time of nitration.

---

* The nitro-body was not specially dried for this determination.

Composition of the mixed acid.—The most important factors in this case are the ratio of H_2SO_4 to H_2O, and the percentage of HNO_3 in the once used acid at the completion of the nitration.

The mixed acid first used had a composition corresponding to :—

	Per Cent.
H_2SO_4	78.4
HNO_3	17.0
H_2O (including HNO_2)	4.6
	100.0

Limits of 0.3 per cent. were allowed for each constituent, so that the dilution was, upon occasion, as high as 4.9 per cent.

In practice this acid did not produce setting-points above 76.5° C., and reinforcement of the acid charge was often necessary, even with a fair HNO_3 content of the once used acid, indicating that dilution of the acid at the end of the nitration was too great, *i.e.*, that the ratio H_2SO_4 to H_2O was too small. The composition of the mixed acid was then changed as follows :—

	Per Cent.
H_2SO_4	78.50
HNO_3	17.25
H_2O	4.25
	100.00

The limits allowed in this case were 0.5 per cent. on the H_2SO_4, and 0.25 per cent. on the HNO_3 and dilution. The latter did not now exceed 4.5 per cent., and reinforcement was not so frequently necessary.

Later, a material reduction in the water content was made through the adoption of a mixed acid of composition :—

	Per Cent.
H_2SO_4	79.5
HNO_3	17.8
H_2O	2.7
	100.0

This acid was finally mixed to 0.1 per cent. limits, and the results were unmistakable; reinforcement was rarely necessary and setting points up to 77.7° C. were readily attained.

In November, 1918, an experimental run was made, using a mixed acid of even lower dilution, viz. :—

	Per Cent.
H_2SO_4	81.0
HNO_3	17.0
H_2O	2.0
	100.0

With this acid, setting-points were reached even more quickly, and there was clear evidence that still higher setting points could be obtained by its use. The graph, Fig. 33, shows the average setting-points obtained with this and the previous acid. The setting-points were determined every half hour after the attainment of the maximum temperature (110° C.). Further

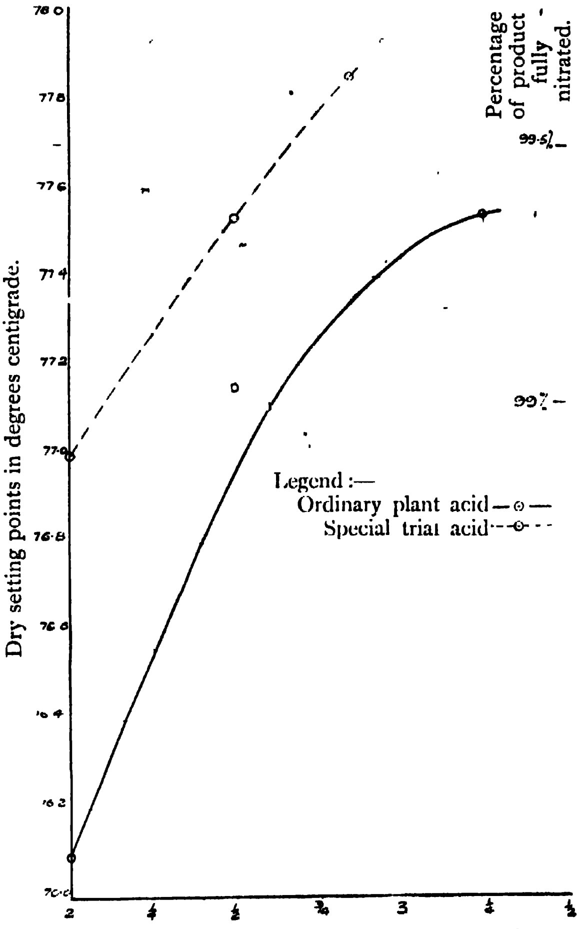


FIG. 33.—CURVE SHOWING PROGRESS OF TRINITRATION IN THE FINAL STAGES.

reduction in the water content of the mixed acid provides, therefore, a solution to the problem of more complete and rapid nitration. The practicability of supplying such an acid depends entirely on the supplies of oleum and highly concentrated sulphuric acid available, and on the ability to arrange a convenient and efficient acid cycle generally.

The trinitration is carried out as follows. The acid charge (11,400 lb.) is blown up into the nitrator from the mixed acid egg, and the "dip" taken as a check on the quantity. The stirrer is then started, and the DNT run in. During the whole of the time in which the DNT is being added, it is necessary for the steam coil to be turned on. The temperature is not, however, permitted to exceed 66° C. until all the DNT has been added; usually a little further heating is necessary after all the DNT is in, before this temperature is reached. At 66° C. the reaction commences, and the temperature of the charge is permitted to rise 3° per 10 minutes to 90° C., and then 5° per 10 minutes up to a maximum of 110° C. The last temperature is maintained until the desired setting-point is attained, samples being taken and tested every half-hour after the maximum temperature has been reached. The charge is then cooled to 90° C., and the dilution water added, the temperature being kept between 90° and 95° C. After stirring for a further 5 minutes to ensure thorough mixing, the stirrer is stopped and the charge is run down to the separator at a temperature of 92° C.

It is convenient at this stage to discuss the remaining factors which govern the completeness of nitration, viz., temperature and time of nitration.

Temperature of nitration.—During the last period of operation, the maximum temperature was reduced to 100° C. without any falling off in setting-point or increase in the time of nitration. This step, which was not possible in the case of the original mixed acid (H_2SO_4, 78.4 per cent.; HNO_3, 17.0 per cent.; H_2O, 4.6 per cent.), resulted in a material reduction in the wear and tear on the lead coils, particularly as regards their tendency to collapse.

Time of nitration.—The greater part of nitration takes place during the rise to the maximum temperature, or, more precisely, during the rise from 66° to 93° C. "Stewing," *i.e.*, maintaining the temperature at the maximum, does give a rise of setting-point, but this is negligible after the first hour. Further improvement in setting-point can only be obtained by reinforcement. Experiments were made to cut down the period of nitration by increasing the rapidity of heat-rise. It was found that a heat-rise of 5° per 10 minutes was easily controlled, and gave precisely similar setting-points on the attainment of the maximum temperature. The gain in time due to more rapid heat-rise (about half an hour) was thus an actual gain in the time cycle. The nitration programme (see following page), as modified in the light of this experience, would be as follows: from 66° to 70° C. in 10 minutes, and thereafter an increase of 5° every 10 minutes up to 100° C. maximum.

Further notes on the cycle.

Acid charge.—No special precautionary measures are necessary except when charging the nitrator after the replacement of coils, or after other repairs which necessitate removing the lid. Under these circumstances, before commencing the nitration, the acid charge is heated to 70° C. for an hour in order to destroy any organic matter, such as wood or cloth, which might possibly remain hidden in the nitrator; the acid is then cooled to 45° C. and the nitration cycle carried out in the ordinary way.

H.M. FACTORY, Unit .... **QUEEN'S FERRY.**

TRINITRATION RECORD.

A.N. No. 2.

Pan .... of N'body from Detoluation overleaf
Batch........... Date..

Charges :—
Acid..........................lb. (Dip in Nitrator.........................inches) started at...............................
MNT............................lb (S G.......................) Charge in Separator by ....
DNT dip in Tank ....in. (S.G.........................) Total Time of Operation......................................
Water...........................lb started at.................

During the Mixing of the DNT charge the temperature should finally reach 66° C, but must not exceed it.

	Time Duration. Hrs.	Mins.	Time. Hrs.	Mins.	Temperature due °C.	Temperature observed °C.	R.P.M. of stirrer.	Reports, Notes, etc.
Running in DNT. up to 66° in 30 mins.	0	0			—			Drowning line tested by
		5			45			
		10			50			3-way Cock turned after greasing by
		15			52			
		20			58			Lubricators given ½-turn by
		25			62			
		30			66			
		35						
		40						
	0	0			66			**CAUTION.**
		10			69			**KEEP**
		20			72			**LIDS**
Heating period 66°—90° in 1 hr. 20 mins.		30			75			**ON**
		40			78			**FUNNELS.**
		50			81			
	1	0			84			
		10			87			
		20			90			
Heating period 90°—110° in 40 mins.		30			95			
		40			100			WET SETTING POINTS.
		50			105			
	2	0			110			
		10			110			
		20			110			
		30			110			
Heating period constant at 110° C.		40			110			
		50			110			
	3	0			110			
		10			110			
		20			110			
		30			110			

Signatures
Operative beginning...........................
Handed over to ....

Running in the DNT.—Using the high gravity nitro-body ("DNT") described above, the addition could be effected as rapidly as the delivery-lines would permit, since there is no appreciable reaction until a temperature of 66° C. is reached.

When it is necessary to start up a house which has been shut down for repairs, acid-MNT of specific gravity 1.19 is charged direct to the nitrators, and under these circumstances precautions are necessary in view of the lower temperature at which reaction commences. The temperature in this case is kept below 51° C. until all the MNT has been added, and is then allowed to rise 3° per 10 minutes to 66° C., after which the normal programme is adhered to.

Temperature rise.—The temperature is usually easy to control. A single coil (No. 2) suffices for this purpose, and is usually in operation for about 15 minutes. At 85° C. a little steam is required and the steam coil is turned on for about half a minute. The temperature then rises to 93° C., after which heating is again necessary in order to bring the temperature up to the maximum. Occasionally, the temperature of a charge gets out of control. This may be due to several causes; (a) through allowing the temperature to rise too rapidly in the early stages whilst using a low gravity nitro-body. This has already been referred to, and no trouble on this account is experienced with high gravity DNT. (b) Leakage of a steam coil; this is revealed by the "bubbling" noise made by the escape of the steam into the charge. (c) Leakage of a cooling coil when working with pressure water; this is indicated by excessive fuming, and can be confirmed by testing the coils in the manner described on page 55. (d) Blockage of the coil outlets or failure of the vacuum. To guard against failure of the water supply sight glasses have been provided at the inlet of each coil (see page 53).

In the case of the temperature getting absolutely out of control, a charge is "drowned" when the temperature reaches 130° C.

Determination of setting point.—When all four nitration sets are in full operation, the output of each nitration house is approximately 25 tons per diem, which calls for the estimation of as many as 70 setting-points per day. It was therefore necessary to devise a simple but accurate method of determination, which could be carried out by the operatives in the nitration house. The first method employed, which proved insufficiently accurate when working to high setting points, was known as the "cold wash method," and was carried out as follows.

Samples of TNT were taken from each nitrator as soon as the nitration charge had reached a temperature of 100° C. The TNT, which carried in solution considerable quantities of nitric and sulphuric acids, was poured into cold water contained in an earthenware mug, the contents of which were stirred vigorously with an aluminium rod. After washing the pellets three times with cold water the liquid was decanted from the pellets, and the TNT transferred to a test-tube, melted, and allowed to cool. Whilst cooling, the TNT must be thoroughly stirred; this is effected by means of a thermometer, and the temperature recorded by the latter is closely observed. The TNT super-cools several degrees below the setting point.

and then, as solidification takes place, the temperature rises until a maximum is obtained. The maximum temperature recorded is known as the wet setting point (W.S.P.) of the sample. The W.S.P. must not be below 72.5° C., in order that the product shall have a dry setting point (D.S.P.) of 76° C. as required by specification. Results from this test are liable to variation unless standard conditions are observed, and a considerable depression of the setting point occurs if much free nitric acid is present in the once used acid from which the TNT has separated, due to the high solubility of nitric acid in TNT (the setting point as determined by this method is depressed approximately 0.5° C. for every 1 per cent. of HNO_3 in the crude TNT). As already pointed out, the nitration cycle is arranged so that the nitric acid content of the once used acid is as constant as possible, viz., 2.0-2.5 per cent., and under these conditions the test is fairly satisfactory in working to a setting point of 76° C.

With the introduction of the sulphite washing process for the production of TNT with setting points above 76° C., it was necessary to exercise a very careful control on the setting point of the crude material leaving the nitration house, and the wet setting point test described above, although sufficiently sensitive when working to a dry setting point of 76° C., had to be modified accordingly. The cold water wash was therefore discarded because of the varying acidity of the resulting pellite, and the alternative test, which was ultimately introduced throughout, consists of a "hot wash" method. In carrying out the latter the TNT is washed with successive quantities of boiling water, the TNT being kept in a molten state, and beaten up thoroughly with an aluminium stirrer consisting of a perforated plate, 2½ inches in diameter, fixed to a handle. About half a dozen washes remove all traces of acid, this point being indicated by a change in the colour of the wash-water from a yellow to a reddish tint. The setting-point of the wet, acid-free product is then determined as before.

The difference between a wet setting-point (W.S.P.) thus determined, and the final dry setting-point (D.S.P.) is 3.7° C. It may be stated in this connection that of this depression only 3.55° is due to water. This has been shown by repeated tests, starting with dry TNT and melting this in water for a wet setting point determination. The remaining depression of 0.15°-0.20° C. appears to be due to the presence of tetranitromethane and other volatile oxidation products.

Dilution.—The dilution charges added at various stages during the trinitration cycle are as follows:—

To nitrators	1360 lb.
To detoluators	400 lb.*
To super-detoluators	480 lb.
Total	2240 lb.

---

* In the houses equipped with preliminary washing plant, the only dilution water added to the detoluators is the wash-water from the preliminary washer.

This yields a spent acid of the following composition :—

	Per Cent.
H_2SO_4	71.2
HNO_3	trace
HNO_2	3.0
Organic matter	0.4
H_2O	25.4
	100.0

Two factors determine the strength of the spent acid :—

(1) *The need for an easily denitrated acid.*—Nitroso-sulphuric acid decomposes completely only at a strength below 70 per cent. With steam

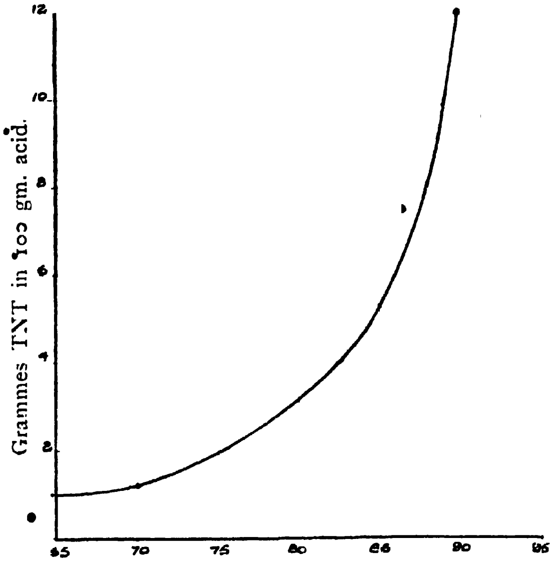


FIG. 34.—SOLUBILITY OF TNT IN SULPHURIC ACIDS OF VARIOUS STRENGTHS AT 90° C.

denitration, the H_2SO_4 content of the spent acid may be permitted to rise as high as 73 per cent. without affecting the ease of denitration.

The increased strength of the spent acid gives a corresponding higher strength in the denitrated acid, the advantage of which is felt at the denitrators.

(2) *The solubility of the nitro-bodies.*—Prior to the introduction of super-detoluation, when DNT was present in the spent acid to an appreciable extent, it was both economical and desirable to produce as weak

a spent acid as possible. With the introduction of super-detoluation, and the elimination of all nitro-body but MNT (which is recoverable), this factor is not so urgent, and latterly the strength of the spent acid has been permitted to rise as high as 73 per cent. H_2SO_4, yielding a denitrated acid of approximately 68 per cent. H_2SO_4. See Figs. 32 and 34.

SEPARATION.

The charge of mixed acid and TNT is allowed to stand in the separator for a period of 30 to 40 minutes. This allows ample time for separation, which takes place without difficulty. It is necessary to use the steam coil at intervals if the charge remains in the separator more than an hour.

PRELIMINARY WASHING.

It will be seen that appreciable quantities of nitric and sulphuric acids remain dissolved in each batch of TNT from the separator, and the following procedure has been adopted with a view to recovering the bulk of this acid before the TNT leaves the nitration house. TNT from the separator sight-box is run into a washing vessel constructed of acid-resisting material (such as silicon-iron), and is there washed molten with hot water with steam agitation. The wash-water is then used for dilution purposes in the detoluator (see page 65), and contains approximately 14.0 per cent. of HNO_3, representing a recovery of 90 per cent. of the nitric acid dissolved in the TNT. Using a hot water charge of 400 lb., the weak acid resulting from the wash contains on an average 14 per cent., or 65 lb. of nitric acid per ton of TNT, representing a saving on previous nitric acid consumption of 3 per cent. In addition to the saving in nitric acid and sulphuric acid, other advantages of preliminary washing are reduced maintenance charges resulting from decreased corrosion of washing plant, tip-wagons, etc., and a reduction in the load on the effluent plant where the TNT wash-waters are neutralised.

The preliminary wash is carried out as follows; 400 lb. of water are introduced into the washer, and the charge of TNT run in from the separator sight-box, the open steam-pipe being turned slightly on. The stirrer is then started and the charge agitated for 5 minutes, after which agitation is stopped, a further 5 minutes allowed for separation, and the wash-water run off to the detoluator. The residual TNT is then discharged to the pelleting vat.

PELLETING.

The pelleting vat is first filled with water until the whole of the sloping base is covered. An even flow of TNT is then allowed to run in, a 1-inch jet of water being directed to a point on the stream of TNT slightly above the surface of the water in the vat. When all the charge of TNT has been pelleted, the water is shut off and the drain-cock opened to allow the water to escape to the launder leading to the recovery and effluent plants. The TNT is then removed to tip-wagons and taken to the washing house.

WASHING.

Acidity of pellite.—TNT *leaving the nitration houses in the form of* pellite has an average composition of

	Per Cent.
TNT	79.5
Total acidity (expressed as H_2SO_4)	1.5
Water	19.0
	100.0

The acidity varies from 1.0-2.5 per cent., and in units where preliminary washing plants are installed this figure is reduced to about 0.5 per cent. In the absence of the preliminary hot-wash, high acidity is caused by one or more of the following causes—(a) faulty separation of TNT from once used acid; this is by far the most serious factor; (b) high HNO_3 content in the once used acid; (c) the use of insufficient water in pelleting; (d) too rapid addition of TNT during pelleting.

An acidity of 1.5 per cent. is made up of approximately 0.3 per cent. of H_2SO_4 and 1.0 per cent. of HNO_3, the remainder being due to organic acids.

Tetranitromethane is a further impurity, which is present in pellite to the extent of about 0.15 per cent.

General aim and methods of washing.—The purpose of the "washing process" is to reduce the inorganic and organic acids to below 0.03 per cent., and to remove the tetranitromethane.

The methods which have been in use up to the present are :—

(1) A combination of hot water washing with successive pelletings. This method is extremely wasteful of heat and compressed air (large quantities of which are necessary in the pelleting stages), and only effected the reduction of the acidity very slowly. The time required for remelting was also considerable, and the whole time cycle for washing was far too long to be practicable.

(2) Discontinuous washing with hot water.

(3) Continuous washing with hot water.

(4) Washing with sodium sulphite solution, for the preparation of high grade TNT.

General.—This was the original process used at Queen's Ferry, and it remained in use until superseded by the continuous and sulphite methods. The equipment of each of the wash-houses working the discontinuous washing process comprised 2 tiers, each of 4 wooden vats, similar to those shown in Figs. 38 and 52. Pellite from the nitration house was tipped into the front row of vats from the tip wagons which run along the front of the building, each vat being fitted with a receiving hopper for this purpose (Fig. 47). The back row of vats was raised on a platform 7 feet above the level of the front vats.

The houses were worked according to one of the two schemes, shown in Fig. 35; (i) the 8 wash-vats were divided into two sets of four, the TNT passing in stages through the four vats; or (ii) the 8 vats were divided into four sets of two, the TNT passing through two only. It was necessary in

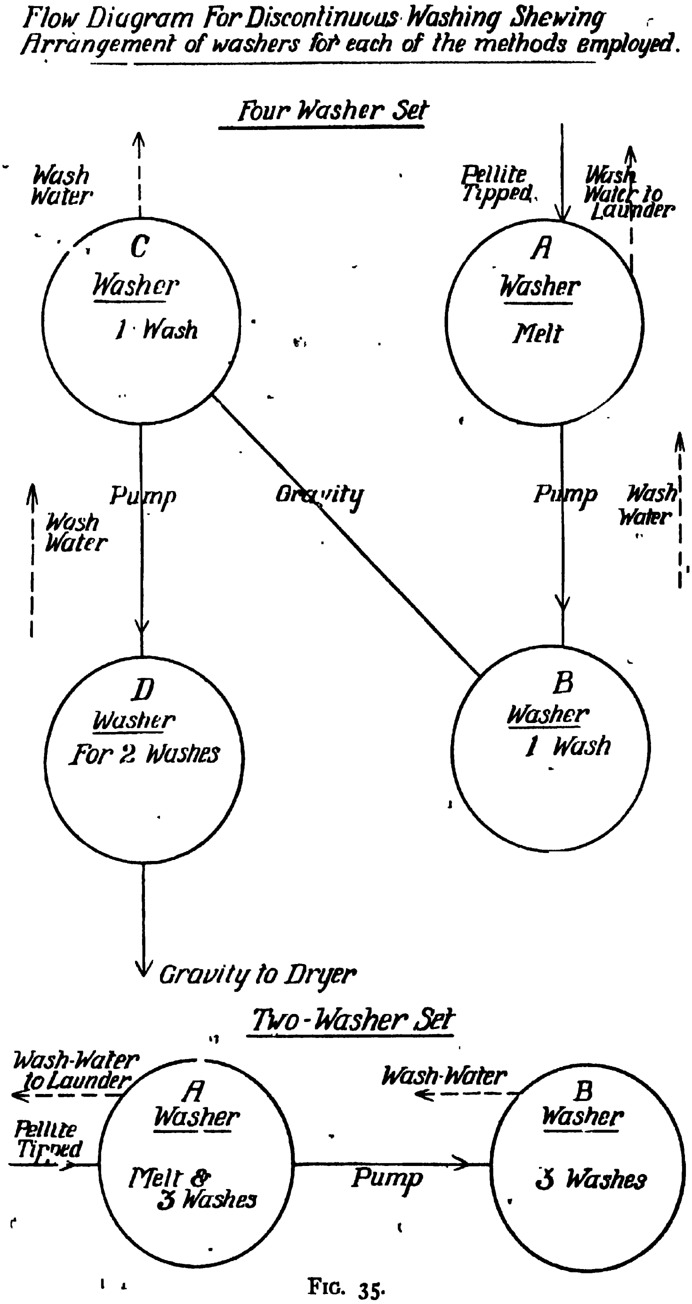
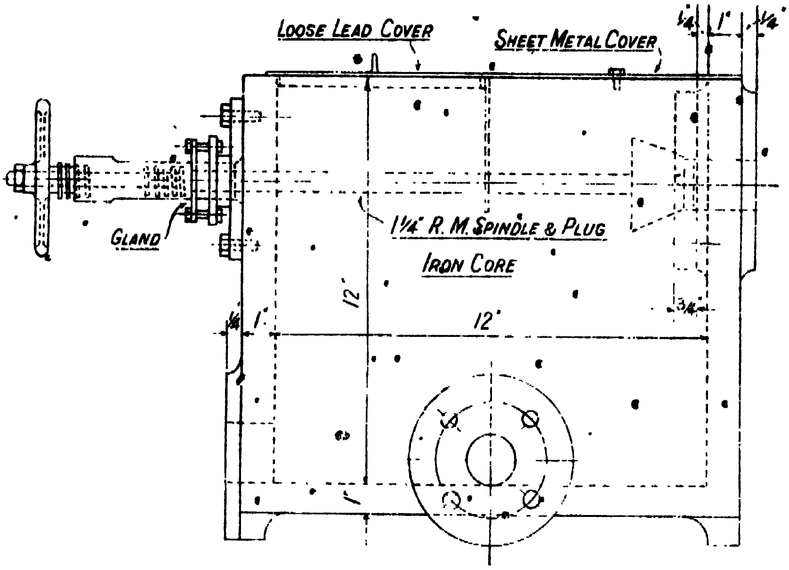
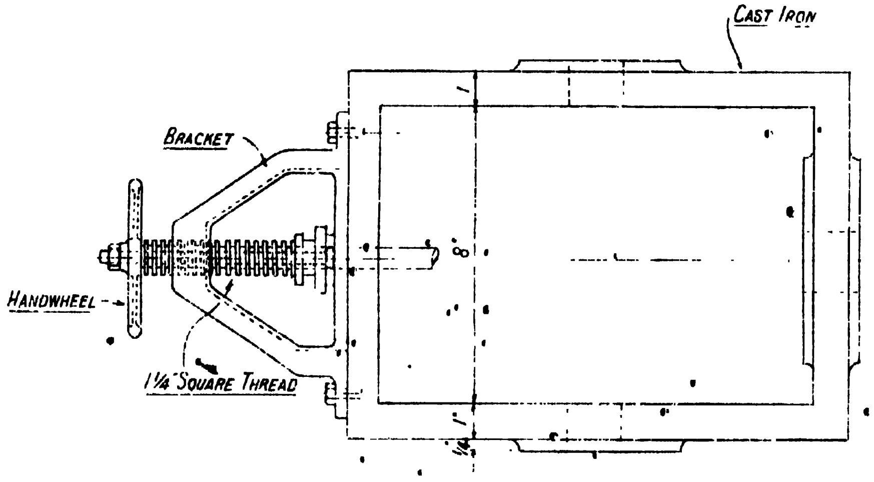

Flow Diagram For Discontinuous Washing Shewing Arrangement of washers for each of the methods employed.

FIG. 35.



— ELEVATION —

ALL FACINGS 6½" DIA DRILLED FOR
FOUR ⅝" STUDS 5" PC & 2" OPENINGS



— PLAN —

Fig. 36.

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both cases to elevate the TNT from the front to the back washers, and this was effected by means of $8\frac{1}{4}$-inch impeller centrifugal pumps.

Wash-vats.—Each wash-vat was fitted with (i) a side-draining box, by means of which the water was run off from above the level of the TNT. This was done after each wash, and also before any TNT was run off or pumped away. The original boxes were of lead, but this metal was replaced by cast iron as renewal became necessary. The lead sight-box on the "A" vat was rapidly corroded, but this material withstood the acidity of the water from the others; the temperature, however, tended to make the lead collapse. (ii) A sight-box fitted with a "bayonet" valve (the general arrangement of which is shown in Fig. 36), and run off pipes for water and TNT to the pump. Originally no sight-box was used, and the TNT was fed through a cast iron steam-jacketed cock direct to the pump. This arrangement was unsatisfactory because it was difficult to detect pump blockages, which are undesirable by reason of the heat generated in the pump by friction; also the efficiency of washing was impaired by the pumping over of wash-water together with the TNT. (iii) An aluminium cover and a vertical sheet iron ventilating shaft, about 12 inches in diameter, which discharges into the atmosphere. (iv) Steam pipes; these were originally of lead, and their use gave rise to the usual emulsion trouble. They were later replaced by silicon-iron and, in the less acid washers, by cast iron.

The equipment of the house was completed by a storage tank for hot water, similar to that described on page 83.

Process.—A charge of boiling water was run into the "A" vat from the hot water tank or "B" vat, as the case may be, this charge being equal in volume to that of the TNT to be washed, *i.e.*, approximately one ton. The TNT charge was then introduced (by pumping, gravity, or, in the case of pellite by tipping) the steam being full on to avoid blockage of steam pipes. The only mixing effected was by means of high pressure live steam (100 lb. per sq. in.), and this was continued for 10 minutes (or for 20 minutes in the case of the pellite, which requires to be melted). The steam was then shut off, and an atmospheric cock fitted to each steam-pipe prevented the sucking back and solidification of TNT in the steam-pipes. Twenty minutes settling was then allowed, after which the water was side-drained, and the TNT run by gravity or pumped to the next vat, unless a further wash had to be given in the same vat, in which case the fresh water was run in immediately. Washes were continued until the wash-water was neutral to service litmus paper. This is found to yield a product with an acidity (calculated as H_2SO_4) of below 0.03 per cent.

The water consumption per ton of TNT in the two schemes of working was:—

(a) In four-washer scheme: reckoning 150 gallons per wash per ton of TNT, and using fresh water for all washes, 750 gallons of water were required. Provided, however, that wash-water from the "B" and "D" washers is utilised in the "A" and "C" vats, this quantity can be reduced to 450 gallons.

(b) In the two-washer scheme: this was only in operation for a short time its main advantage being rapidity of working and less pumping of the TNT. Six washes were usually necessary, which is equivalent to 900 gallons of water. This quantity could be cut down to about 600 gallons, by a further use in the "A" vat of one or two charges of water from the "B" vat

The capacity of a wash-house operating the discontinuous process was 30 tons of TNT per day.

It is necessary, particularly in the two-washer system, to adjust the charge of TNT so as to leave as little water as possible in the washer after side-draining, and also to allow sufficient time for settling.

The arrangements for conducting the wash-waters from the vats to the effluent launder were similar to those used in the continuous washing process. Cast iron pipes are most satisfactory for this purpose, since earthenware lines and joints crack owing to the temperature.

CONTINUOUS WASHING.

General.—This process (see page 13) was developed as a result of preliminary laboratory and semi-works scale experiments carried out at H.M. Factory, Oldbury.

TNT in the molten state and hot water are made to flow from one vessel to another in contrary directions. Each vessel is fitted with mechanical stirring gear and with a subsidiary separating vessel in which the materials separate, and from which they pass to the succeeding washing vessels in opposite directions. This method of washing has resulted in considerable economies in steam and water consumption, and has been extended to all washing houses not using the sulphite process. The essential points in the construction of the plant are indicated diagrammatically in Fig. 37.

As installed at Queen's Ferry, the plant comprises two melt vats into which is tipped and melted the pellite from the nitration houses; a TNT storage tank, into which is pumped the melted TNT from the melt vats ready for delivery to the washer; a storage tank for hot water, with steam-heater and feed arrangements for supplying the hot water to the washing plant; the continuous washer proper, which is fed with molten TNT and hot water from opposite ends; a separator, into which the washed TNT overflows, and from which it passes to the drying plant; and a cooling launder along which the wash-water is made to flow, after leaving the washing plant, on its way to the recovery pits and effluent treatment plant.

Melt vats.—These are two of the original "A" and "B" discontinuous process wash-vats. As shown in Fig. 38, they are semi-cylindrical in shape, 8 ft. 6 in. long × 7 ft. wide × 4 ft. deep, of 700 gallons capacity, and are constructed of 3-inch pitch pine lined outside with heavy sheet lead. The interiors are lined throughout with acid-resisting slabs, 3 inches thick, jointed with acid-resisting cement, and this lining has been very effective in prolonging the life of the vats.

The heating pipes consisted originally of 2-inch lead pipes perforated with ½-inch holes, the TNT being heated by live steam. These pipes had only a very limited life (150 tons, or 4 days' average running), and caused a great deal of trouble owing to the formation of lead sulphate sludge, which

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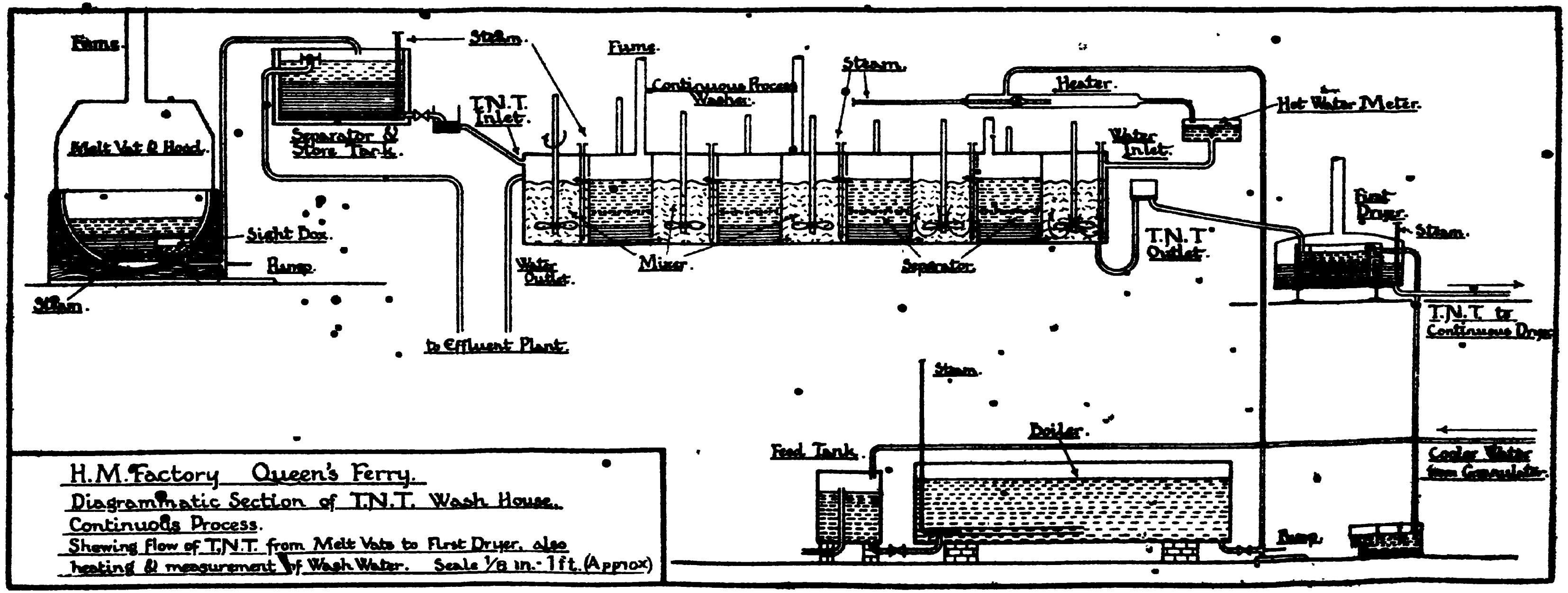
Fume.
Melt Vat & Hood.
Sight Box.
Pump.
Steam.
Steam.
Separator & Store Tank.
T.N.T. Inlet.
to Effluent Plant.
Fume.
Continuous Process Washer.
Steam.
Heater.
Hot Water Meter.
Water Inlet.
Water Outlet.
Mixer.
Separator.
T.N.T. Outlet.
First Dryer.
Steam.
T.N.T. to Continuous Dryer.
Steam.
Boiler.
Feed Tank.
Pump.
Cooler Water from Granulator.
H.M. Factory Queen's Ferry.
Diagrammatic Section of T.N.T. Wash House.
Continuous Process.
Shewing flow of T.N.T. from Melt Vats to First Dryer, also heating & measurement of Wash Water. Scale 1/8 in.-1 ft. (Approx)

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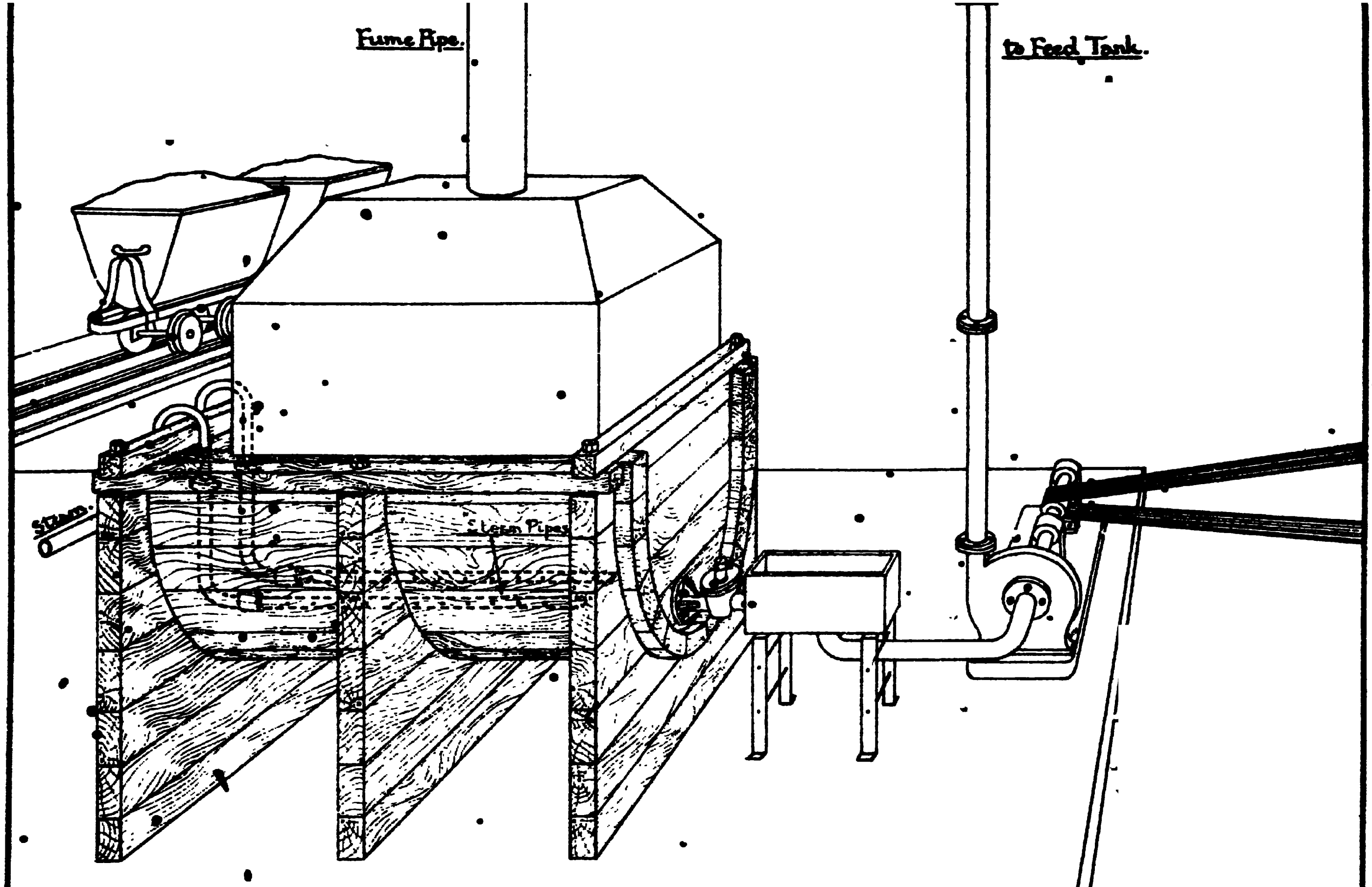
Fig. 37.



H.M. Factory Queen's Ferry.
T.N.T. Section. Continuous Washer.
Shewing arrangement of Melting Tub, Sight Box
& Pump to Feed Tank.

Fig. 38.

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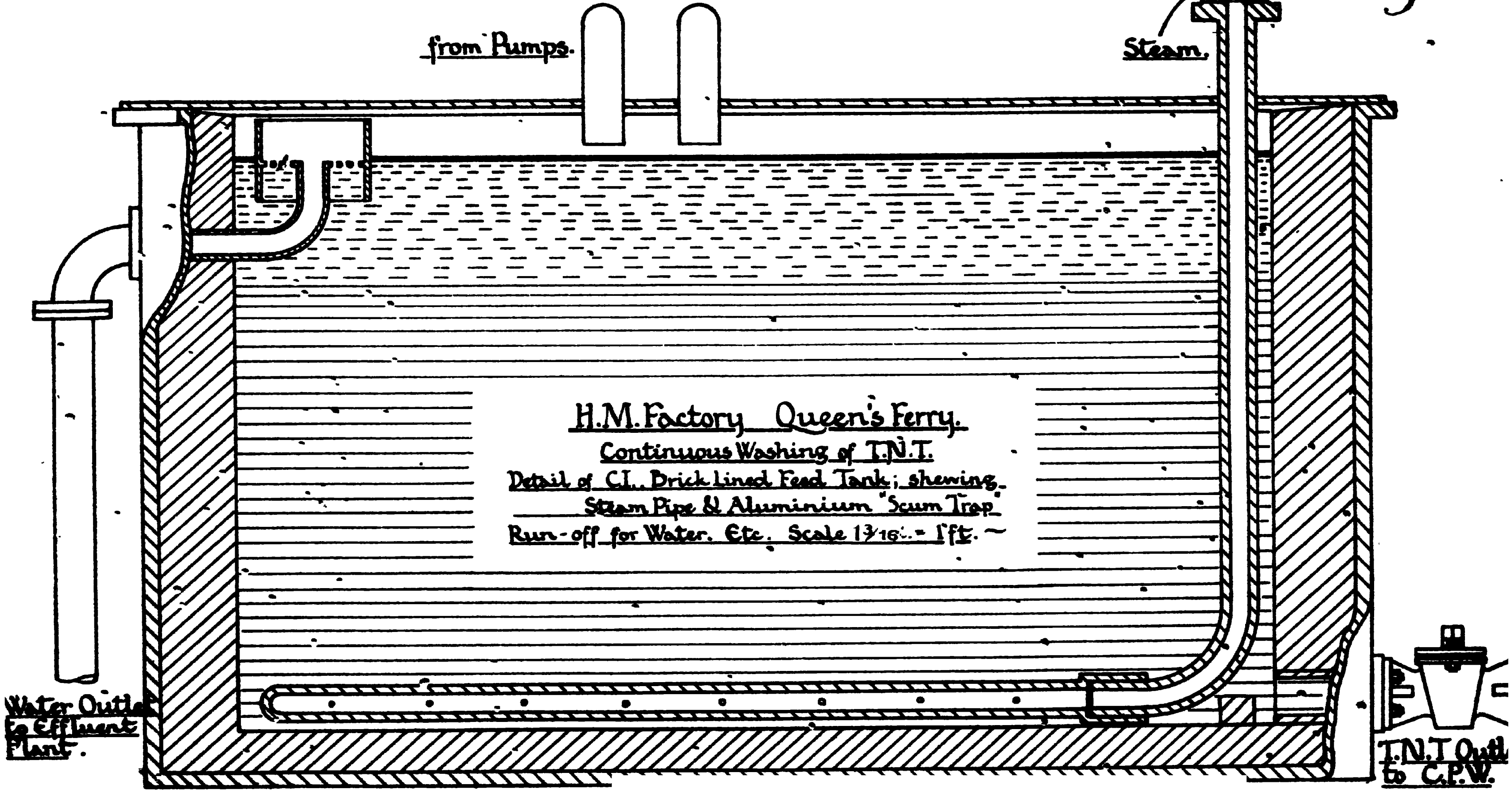
Fig. 39.


from Pumps.
Steam.
H.M. Factory Queen's Ferry.
Continuous Washing of T.N.T.
Detail of C.I. Brick Lined Feed Tank; shewing Steam Pipe & Aluminium "Scum Trap" Run-off for Water. Etc. Scale 1 3/16" = 1 ft.
Water Outlet to Effluent Plant.
T.N.T Outlet to C.P.W.

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gave rise to emulsions in the continuous washer, resulting in bad separations throughout the plant. The steam pipes at present in use are of 2-inch silicon-iron, with eight ⅜-inch holes, and are jointed with lead. After treating 2,600 tons of TNT these pipes are still in good condition, and the emulsion trouble is no longer encountered.

It is necessary to provide a grid to protect the outlet from the vat, and no suitable material has yet been found for this purpose. Aluminium is soon corroded, the corrosion factor being as high as 80.* Lead is the most satisfactory metal which has been tried up to the present, a lead grid lasting about 2 months, but it is probable that silicon-iron would be most suitable, as the corrosion factor for this material is only 1.6.

Connections between melt vats and TNT storage tank.—The outlet from each melt-vat is through a 2-inch silicon-iron cock and a sight-box of the same material to a centrifugal pump (8¼ inch impeller) which elevates the liquid TNT about 12 feet to the store tank through a 2-inch silicon-iron pipe. This arrangement is the most unsatisfactory feature in the design of the plant. With the system of melting the TNT by live steam alone, *i.e.*, without the addition of an initial charge of hot water, the acid water resulting from the condensed steam has the following acid content:—

	Per Cent.
H_2SO_4	3.0
HNO_3	4.9

Weak acid of this composition has naturally proved highly corrosive to metals other than silicon iron. The outlet cock has lasted well, no renewals being necessary. The sight-box is always kept full of hot water (this being due to the drainage from the delivery pipe from the pump). but despite this fact its life has also been satisfactory. Cast-iron sight-boxes were previously tried, with an average life of about one week.

The pump is a cast iron centrifugal with 8¼ inch impeller. A steam-jet is fitted into the air release of the pump and is kept open when the pump is not in use, the steam making its exit through the water in the sight-box. The pump impeller requires renewal once a fortnight, and the pump case once a month. Chrome steel has been suggested as a suitable material for these pumps, but the suggestion has not yet been given effect.

TNT storage tank.—The storage tank, Fig. 39, consists of a cast iron tank, 6 ft. 3 in. diameter × 1 in. thick, lined with acid-proof brick set in acid resisting cement, the capacity is just over 2 tons. An overflow is provided for the wash water pumped up with the TNT, which is connected to the launder by a 2-inch silicon-iron pipe. A skimming arrangement prevents the scum which rises to the surface of the TNT from blocking the launder pipes. The most satisfactory form of skimmer consists of a 2-inch silicon-iron elbow; the aluminium skimmer shown in Fig. 39 proved unsatisfactory on account of its rapid corrosion.

---

* The corrosion factor expresses the number of grammes of metal dissolved per hour per square metre of metallic surface.

The TNT is heated by a straight silicon-iron steam pipe, but heating is only necessary when the plant is temporarily held up. The outlet is through a 2-inch silicon-iron cock, by means of which the rate of flow of the TNT to the continuous washer is controlled. A brick ledge, 4½ inches high, is built round the outlet on the inside of the store tank, and serves to retain any heavy lead sulphate sludge in the store tank when lead coils are in use. The outlet cock delivers into an earthenware sight-box feeding a 3-inch earthenware pipe, supported by a channel-iron, which discharges into the first agitator chamber of the continuous washer.

Continuous washer.—The continuous washer consists of nine sets of two compartments each, one of which is a mixing chamber and the other a separating chamber. The washer is constructed of cast iron plates bolted together, the joints being made with red lead and a double strand of lead wire; the division plates between the mixing and separating chambers are loosely held in slots formed in the cast iron cross-plates. The whole thus forms a rectangular tank, 27 ft. long × 4 ft. 6 in. wide × 4 ft. 3 in. deep (Figs. 40 and 41). The width of the washer is distributed between mixing and separating chambers in the ratio of 2 to 1.

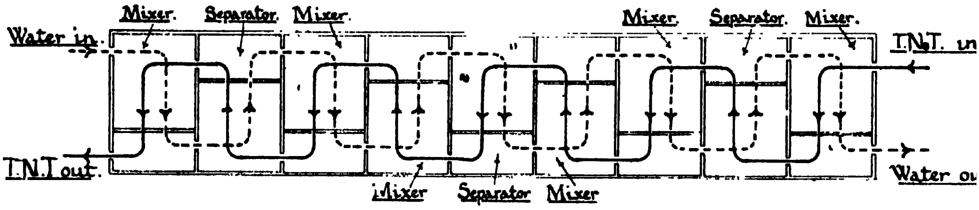

FIG. 42. DIAGRAM SHOWING FLOW OF WATER AND TNT IN CONTINUOUS WASHER.

Each of the plates separating the mixing and separating chambers is perforated by a row of twelve 1-inch holes at a distance of 16 inches from the bottom of the washer. Through these holes the emulsion of TNT passes from mixer to separator. Thus, to maintain the flow it is necessary for TNT and water to enter each mixer and leave each separator by holes in the cross plates dividing the sets. The TNT outlets are 2½ inches in diameter and are situated at the bottom of the washer; the water outlets are of the same diameter, but are placed 30 inches from the bottom. Fig. 42 indicates the directions of flow of TNT and water through the washer. The TNT outlet is taken from the bottom of the last separator chamber and is led by an inverted siphon to the cast iron sight-box, the bottom of which is 23 inches above the bottom level of the washer. This arrangement ensures an overflow of TNT through the sight-box when the dip of TNT in the last separator exceeds 8 inches. Each mixing chamber is fitted with a stirrer, 20 inches in diameter, and a steam-pipe with perforations at its lower end. The four steam-pipes nearest the water outlet are of silicon-iron, and the five remaining ones of wrought iron; cast iron, however, would be quite satisfactory throughout. Drain outlets are provided at the bottom of each chamber, and these are closed with lead blank flanges during the

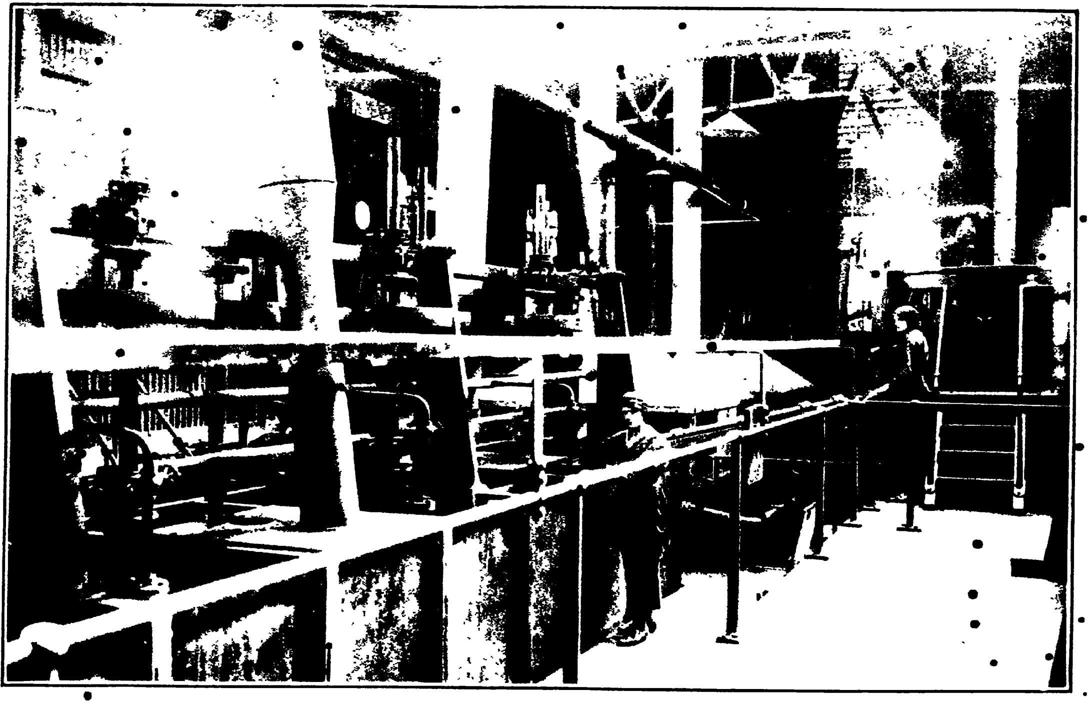

Fig. 40.

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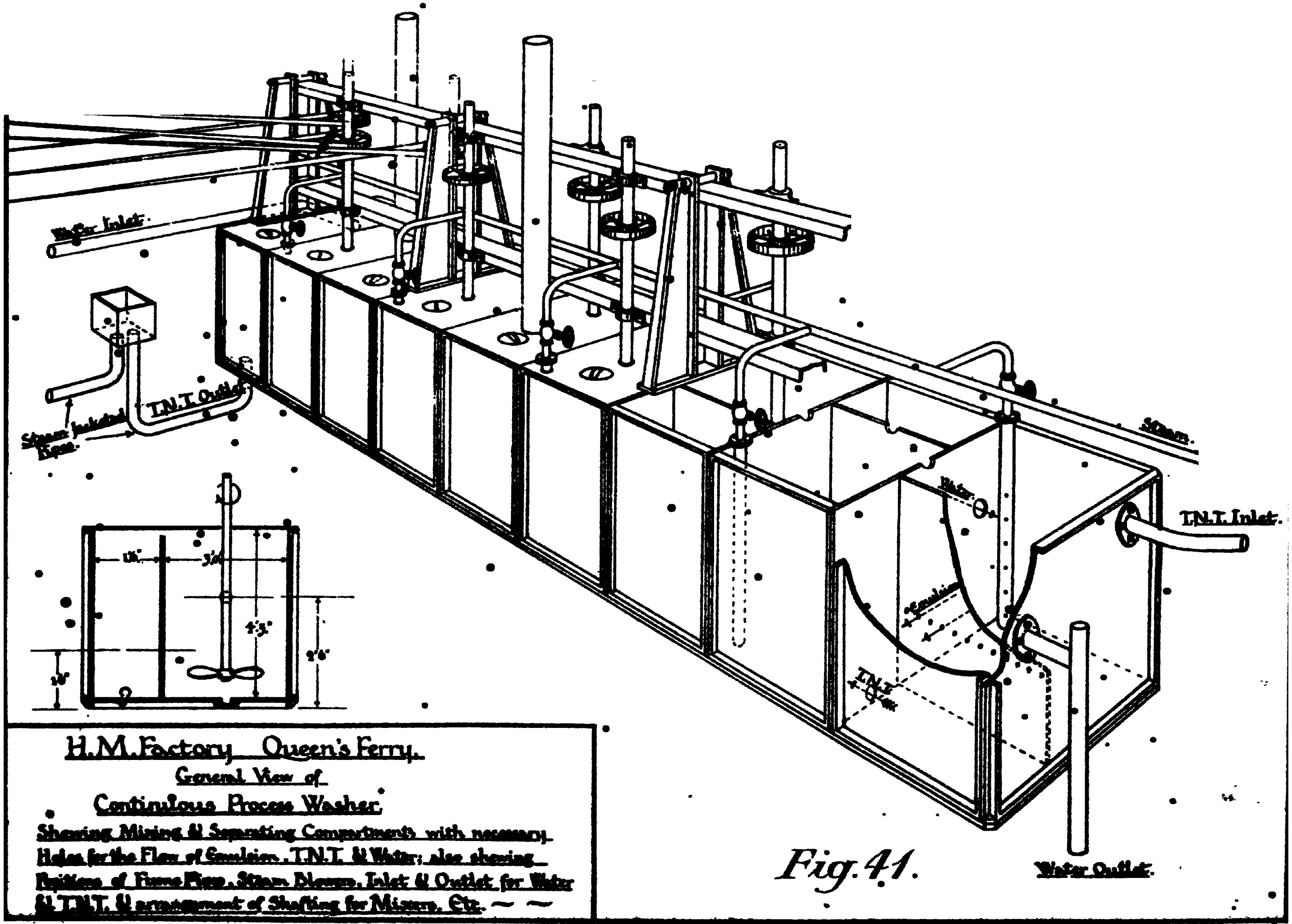

Fig. 41.

H.M. Factory Queen's Ferry.
General View of
Continuous Process Washer.
Showing Mixing & Separating Compartments with necessary Holes for the Flow of Emulsion, T.N.T. & Water; also showing Positions of Fume Pipes, Steam Blowers, Inlet & Outlet for Water & T.N.T. & arrangement of Shafting for Mixers, Etc. ~ ~

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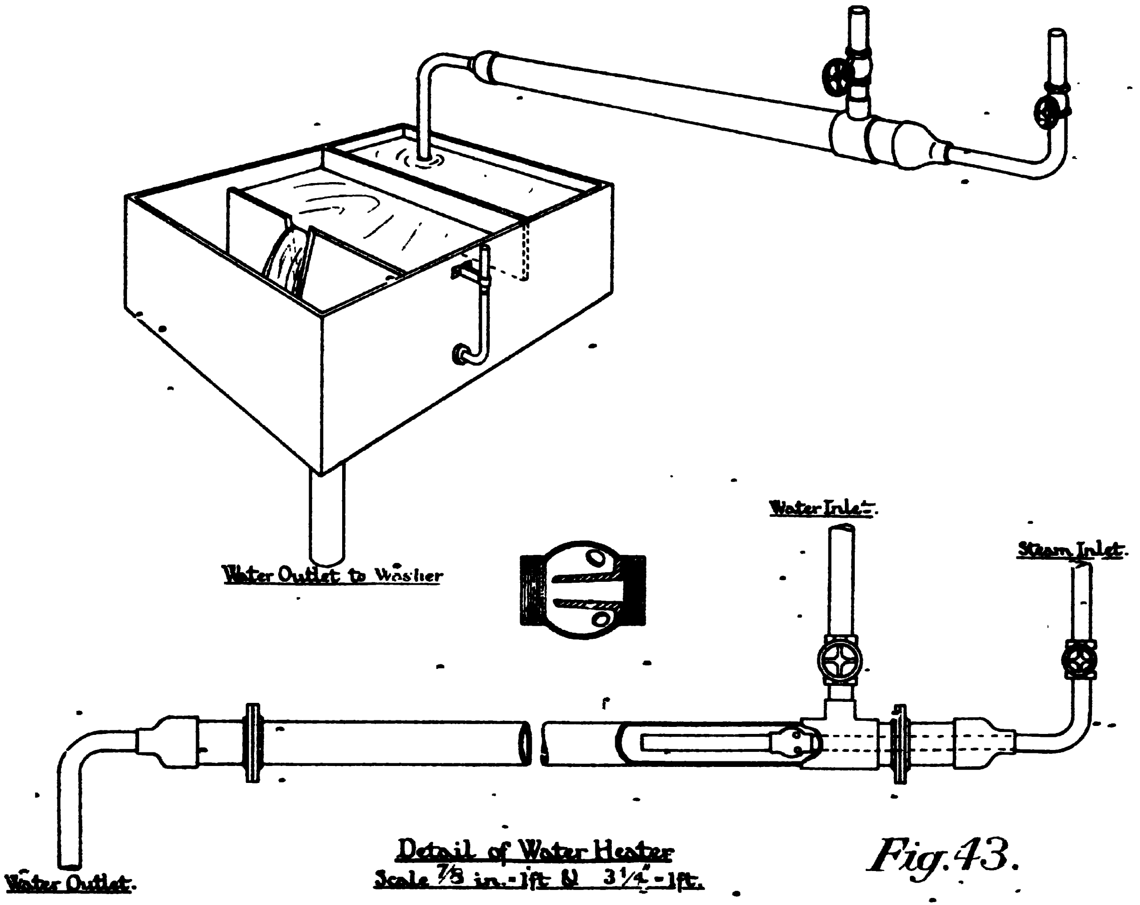
H.M. Factory Queen's Ferry. T.N.T. Continuous Washing Process.

General View of Water Heater & Measuring Box.



Detail of Water Heater
Scale 7/8 in.-1ft. & 3¼"-1ft.

Fig. 43.

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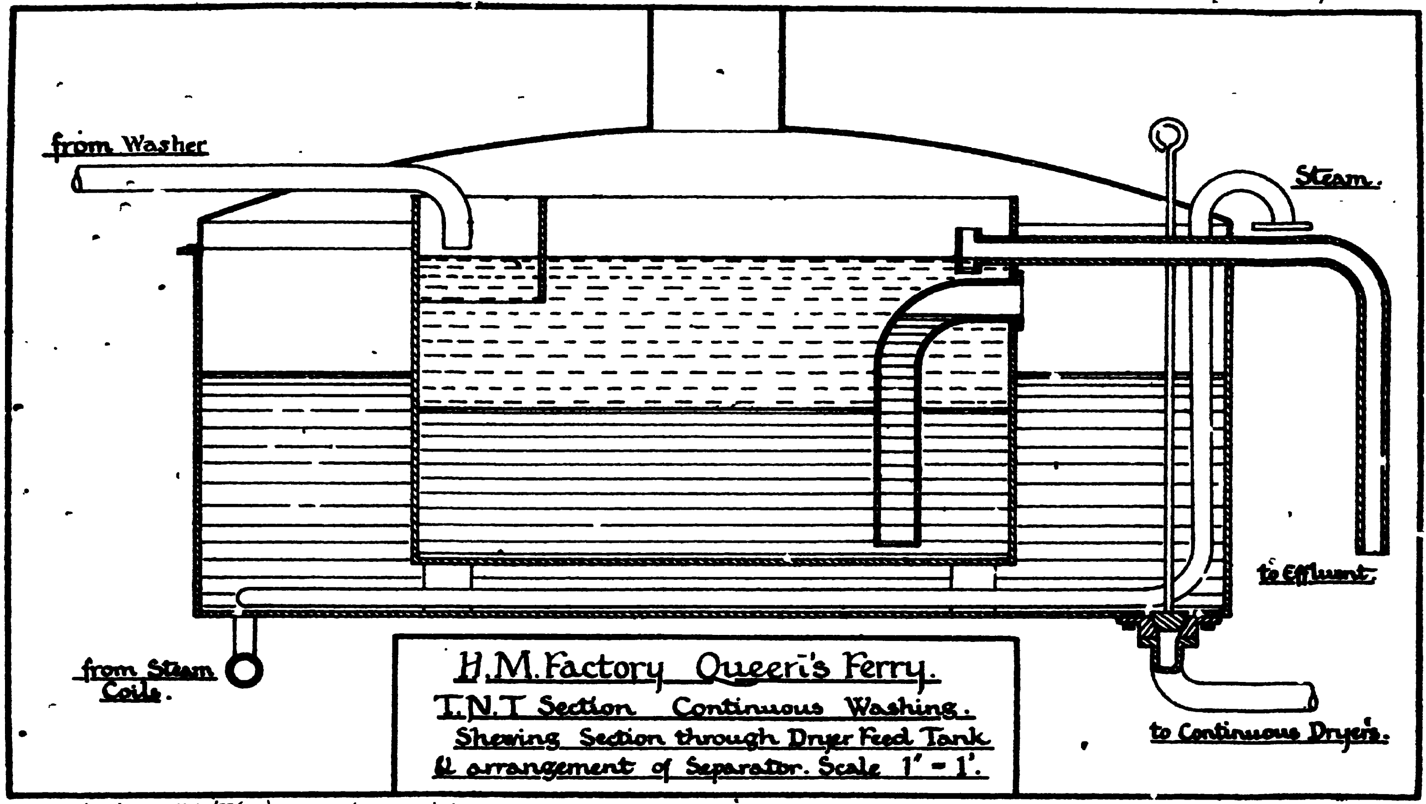

H.M. Factory Queen's Ferry.
T.N.T Section Continuous Washing.
Shewing Section through Dryer Feed Tank
& arrangement of Separator. Scale 1' = 1'.

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Fig. 44.

period the washer is in use. One or more of these is pierced whenever it is necessary to drain the washer. Thermometers are placed in every third chamber.

Hot water feed arrangements.—These consist of a boiler, 7 ft. diameter x 25 ft. long, of about 5,000 gallons capacity, in which the temperature of the water is raised to 85°C.; the feed water is supplied through a float-valve from a supply tank fed from the ordinary supply, and into which also passes the warm water from the granulator roll (see later) at a temperature of about 30° C. The boiler is heated by steam direct from the main, and the steam exhausts from the driers and steam mains also discharge into it. The water is supplied to the washer by means of a centrifugal pump coupled direct to a motor. A secondary heater of the Pemberly type is inserted between the pump and a water measuring box, by means of which the temperature of the water is still further raised. The rate of flow of water passing into the washer is controlled by a V-notch inserted in the measuring tank (Fig. 43). From the measuring tank the water flows into the washer through a 2-inch wrought iron pipe.

Separating tank.—TNT leaving the washer passes by means of a 2 inch wrought iron pipe to a separating tank situated inside the feed tank to the driers. The separator (Fig. 44) consists of a wrought iron tank, 3 ft. 6 in. diameter x 2 ft. 3 in. deep. It is provided with a baffle against the incoming TNT, a water overflow 5 inches from the top, and a TNT outlet 4 inches below the latter, which is fitted with a pipe that reaches almost to the bottom of the tank.

Effluent launder.—The wash-water is conveyed by means of a cast iron pipe to a wide shallow launder constructed in acid-proof brick, the general construction of which is shown in Fig. 45. The wash waters are cooled whilst flowing through this channel, and deposit, before reaching the main effluent drain, any TNT which may be held in suspension or separated from solution. The deposit is removed at intervals and the TNT, the greater part of which settles out in the first half of the channel, is returned to the washers by means of the tip wagons used for pellite. The deposit in the second half of the channel is removed to the recovery plant, and that in the rest of the launder is swept into the effluent drain and thence into the catch-pits, from which it is removed at intervals to the recovery plant.

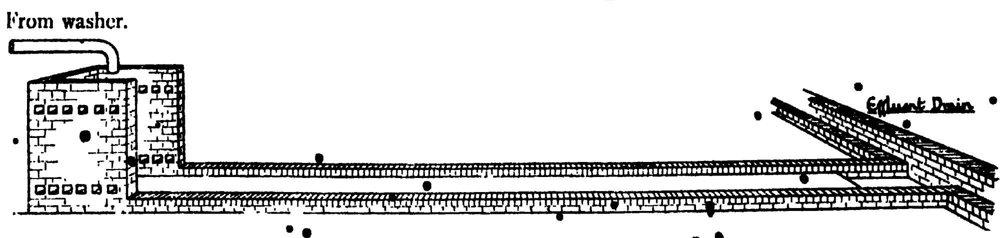

FIG. 45. PERSPECTIVE DIAGRAM OF ACID PROOF BRICK LAUNDER CARRYING WASH WATERS TO EFFLUENT DRAIN.

Launder = 150 ft. long, 4 ft. 6 in. wide, and 9 in. deep.

Process.—The charge for each melt-vat consists of 3 tip-wagon loads, *i.e.*, about 27 cwt. of pellite. This is tipped into the vat and the steam turned on, the charge melting in about 20 minutes. It is customary to keep one batch melted as a stock of TNT supplementary to that in the feed tank, since the capacity of the latter only just exceeds one charge and it is essential to keep a further charge in readiness in order to ensure the flow of TNT being continuous. From the feed tank onwards the discontinuous process gives place to the continuous process. A fresh charge of TNT from the melt-vats is pumped into the feed tank as soon as the dip of TNT falls to 4 inches, and the rate of flow to the washer is regulated by means of the outlet cock. During normal running the necessary control is as follows:—

(1) The temperature of the wash water entering the washer must be kept above 90° C.

(2) The temperature of the TNT in the feed tank must be kept between 80° C. and 90° C.

(3) The temperatures throughout the various compartments should range between 95° C. and 100° C.; this necessitates the use of steam pipes in the agitator chambers, on account of the large loss of heat due to radiation.

(4) The TNT leaving the feed tank sight box is tested hourly for acidity. This usually ranges between 0.10 and 0.20 per cent. In the event of a high acidity, the flow of TNT is checked, and additional water is passed through the washer.

(5) The average dip of TNT throughout the washer is 8 inches, and should not exceed 12 inches. Any banking up of TNT is due to blockage, caused usually by a fall in temperature in some part of the washer.

To close down the process, the TNT melt vats and feed tank are emptied, and the washer kept working with a normal flow of water. The greater part of the TNT is thus made to move along towards the outlet, and steadily overflows; as a rule about one inch of TNT remains in the bottom of the washer. When this condition has been reached, the flow of water and the agitation are stopped, and the washer is emptied by piercing the lead blank flanges which seal the bottom outlets.

To start up the process, the lead blanks are replaced and the washer is then filled up with cold water, the TNT outlet being plugged meanwhile. The steam pipes are then turned on, and at the same time the flow of hot water is started. When the temperature throughout the washer reaches 95° C. the agitators are started and the TNT admitted to the washer, at first slowly, the flow being then steadily increased to the normal (1¼ tons per hour). When the TNT dip in the last separator reaches 8 inches the plug is removed and the TNT allowed to overflow into the sight-box.

Capacity of plant.—The capacity of the washer described above is about 1¼ tons per hour, the largest output attained being 260 tons per week of

168 hours. The overall water consumption per ton of TNT washed is approximately 550 gallons.

A facsimile of the continuous washer log-sheet is reproduced on the following page.

SULPHITE WASHING.

General.—In the latter half of the year 1917, "A" wash-house underwent the necessary alterations for conversion from a discontinuous washing process to a process for sulphite purification. The new plant commenced operations early in January, 1918, and continued to run with little intermission until November of the same year. During this period the process was extended to "B" unit, where, apart from certain modifications to which attention will be drawn, the original procedure was adhered to.

It may be noted that the avoidance of structural alterations in the building somewhat limited the scope of the lay-out. It was found to be necessary to pump the product at one stage, and, partly on safety grounds, it was decided to pump a wet crystalline sludge in preference to liquid TNT. Experience gained in operating the process has indicated that the arrangement adopted was undoubtedly the best which could have been devised in the circumstances. As regards details, however, several alterations were afterwards found desirable, and, in particular, it was found that the crystalliser time-cycle had been underestimated, and to work up the capacity of the other portions of the plant it was necessary to extend this section.

The diagrammatic elevation of the plant shown in Fig. 46 indicates the various stages in the process. The building contains two duplicate sets, each comprising three crystallisers and one each of the various other portions of the plant. These two sets are arranged on either side of the house, and are self contained and isolated from each other.

Preliminary wash- or melt-vat ("A" vat).—It is desirable for a variety of reasons that the crude TNT should undergo a preliminary washing before crystallisation. The main considerations are that the crystallising plant is thereby largely relieved from the corrosive effects attending the treatment of acid TNT, and the objectionable tetranitromethane fumes are largely removed by steaming.

The crude pelleted TNT, after passing the specification test discussed later, is brought into the house, elevated by a lift, and discharged from the tip-wagons direct into the "A" or melt-vats (Fig. 47). It is delivered into an approximately equal bulk of boiling water, and heated by open steam coils, agitation being effected by a cast iron stirrer running at 75 r.p.m. The charge melts completely within ten minutes, and the molten TNT is then washed with four successive changes of boiling water, each wash proceeding with agitation for ten minutes, followed by ten minutes settling and removal of the supernatant wash water. The upper side-draining box by which the wash waters are drained communicates by a cast iron line with a brick cooling launder outside the house (see page 83). Wash waters from the "B" vat (see later) also deliver to this launder. The wash waters from the final-wash or "C" vat are used again in the "A" vat, as they are uniformly free from acidity and already saturated with TNT. The upper side-drain is set 21 inches from the inside bottom level.

H.M. FACTORY, QUEEN'S FERRY.

CONTINUOUS WASHER RECORD.

Unit.. .... 24 hours ending 6 a.m. 191

	No. 1 melt vat	Feed Tank	No. 2 melt vat	WATER FLOW IN. Gallons per hour.			ACIDITIES.				TNT IN SEPARATORS.			
				500	600	700	.010	.015	.020	.025	Feed Tank.	1	3	7
6 a.m.														
7														
8														
9														
10														
11														
12														
1 p.m.														
2														
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2														
3														
4														
5														
6														

Repairs executed :—

General remarks :—

FIG. 47.

To face p. 8

Practice requires, however, a working limit of about 3 inches, so that the depth of the TNT charge is approximately 18 inches, the charge of crude TNT being regulated by dip after melting. The hot water supply is similar to that described on page 83.

Where no preliminary wash is given in the nitration house, the initial acidity of the pellite averages 2 per cent. calculated as H_2SO_4, and the washing programme outlined above reduces this to less than 0.1 per cent. With the installation of preliminary washing plant in the nitration houses, the initial acidity is reduced to 0.5 per cent., and in this case the number of washes given the "A" vat is cut down accordingly.

The washed TNT is run down from the melt-vats to the crystallisers, where the required charge is regulated by "dip," any TNT remaining in the "A" vat forming part of the next charge. Each "A" vat serves three crystallisers, and the charge is run to any desired crystalliser by means of a three way sight-box, which serves as a header to three independent lines, each communicating with a crystalliser. Control of these lines is effected by rubber plugs, held in position between two metal washers, and which fit into a seating where the line takes off from the sight-box. The contents of the vat are discharged into the sight-boxes through a screw valve, which carries a sunk rubber face and bears upon a cast iron renewable seat screwed into a fitting on the vat (Fig. 52).

All the vats are covered with hinged sheet-iron covers, to which are fitted vertical vent pipes which pass through the roof and discharge into the atmosphere.

As might be anticipated, treatment entailing the boiling of dilute acids has a very destructive effect on the plant, and it has been found necessary to modify this portion of the plant in several directions. Lead coils were at first used, but the life of these was extremely short. Later, 2-inch internal diameter silicon iron pipes with ¼-inch perforations were tried, and have proved most successful. On account of the difficulty of making the required length in one piece, these pipes are cast in two pieces, with socket and spigot, the joint being made with well rammed lead. This type has proved so successful that its use has been extended to all vats in the building. Small branch relief valves, with extensions delivering just under the vat lid are fitted immediately below the steam control valve to each vat. When the steam is turned off to allow the charge to settle before draining away the wash water, these relief valves are opened, and thus obviate any disturbance in the vat, which would otherwise occur when the control valve leaks. So far, only one silicon-iron steam pipe has required replacement, after treating approximately 1,500 tons of TNT.

The rubber seating to sight-box valves occasioned much trouble for a time, becoming soft and easily destroyed by the heat and by the necessary pressure required to close the valve. Later, however, more highly vulcanised rubber was used, with excellent results. So far, any replacements have been occasioned by damage to the rubber face by chips of wood, bolts, or other debris.

The wooden false bottom originally fitted in the washer had a very short life, and succeeding replacements met with no better results. Later it was replaced by a lining composed of large slabs of obsidianite set in acid-resisting cement, and this arrangement has resulted in a much increased life of the vat.

Several stirrer blades broke soon after fitting, and it was concluded that this was due to the wedging effect of the blades upon the large lumps of TNT which are occasionally present. The stirrers were accordingly shortened by 6 inches, so as to allow a clearance of 9 inches, and since this alteration was made no case of breakage other than from wear has occurred. The wear is most marked on the face of the blades, which finally snap off when the blade has worn down to about 3/16 inch in thickness; their average life is 500 batches. The stirrers are made with shaft and propeller head separate, in order to permit of the renewal of the latter.

In the plant designed for "B" wash house the 3-way sight-boxes referred to above have been replaced by boxes carrying a fourth line, which communicates with the launder pipe from the upper side-drain. This enables the vat to be emptied for inspection or repair with much greater facility than is possible with the present 3-way type.

The first pair of "A" vats were constructed of pitch pine, and these were used to treat some 1,900 tons of TNT before wearing too thin to support the sight-boxes. They were replaced by a pair of vats made of oak, which lasted only some 200 tons each, after which they collapsed. Replacement had again to be effected in oak, and these were lined with tongued and grooved 1-inch pine boards, but with little success, the acid water finding its way behind this lining and severely affecting the oak. This experience indicates in a most marked degree the superiority of pitch pine for this class of work.

Crystalliser.—The purpose of crystallisation has already been outlined on page 19. Since the degree of purification is largely dependent upon the nature of the crystallised product, it is evident that the formation of a suitable form at this stage is of paramount importance. The problem presents a number of novel features in the matter of plant design, and the first crystallisers installed exhibited some defects. In the subsequent additions to this section of the plant, modifications were experimented with, and finally a type of crystalliser was evolved which proved on the whole quite satisfactory.

It may be advisable to summarise at this stage the more important considerations which arise in this connection. The ideal conditions include :—

(1) The production of a maximum of very fine crystals (under 20 mesh), and an avoidance of pellets and lumps.

(2) Crystallisation and further reduction in temperature to be effected as rapidly as possible.

(3) Rapid and complete discharge of the contents of the crystalliser, so as to leave the latter clean for a fresh charge.

(4) Avoidance of "dead" areas on which TNT will set in hard lumps.

(5) Avoidance of any features tending to break the fine crystals after formation.

(6) Efficient mixing of the charge after sulphiting, to ensure rapid treatment and yield a homogeneous sludge which can be easily pumped.

(7) Avoidance of complicated designs for blades and attachments, in order to permit of rapid replacements as a result of wear or breakage.

(8) Large margin of power, to allow for starting up after accidental stoppages whilst crystallisation is in progress.

An extended series of experiments, carried out at the laboratory at the factory, determined the degree of purification effected and the rate of attack of TNT by sodium sulphite under varying conditions of time, temperature and concentration. As a result of these investigations the optimum conditions were determined to be :-

(1) Reaction temperature, 40°-45° C.*

(2) Ratio of sodium sulphite to TNT, 3½-4 per cent.

(3) Strength of reacting solution, 3-4 per cent. Na_2SO_3.

(4) Time of reaction, ½ hour.

In practice these conditions have given very good results so far as degree of purification is concerned.

Since any excess of sulphite over that necessary for removal of the isomers represents wastage of α TNT, and, since the conditions obtaining on the plant differ in many respects from those in the laboratory, investigation of the lower limits of these factors under process conditions is desirable. For a variety of reasons, however, it has not been possible to conduct experiments on these points. The trouble accompanying the installation of new plant and the operation of an entirely new process with untrained labour occurred in a marked degree, and, combined with the demands of maximum output and the necessity for increasing the crystallising plant itself, it was expedient to postpone any plant research until a more favourable opportunity. Since the theoretical ratio Na_2SO_3/TNT (assuming complete removal of isomers and no α TNT attacked) is under 2.5 per cent., it was considered that the percentage of sulphite employed (4 per cent.) might be capable of reduction without affecting the degree of purification.

At the time when it appeared possible to undertake investigations in this direction, it was decided to make Grade 1 TNT, and, in view of the higher specification for this Grade, the experiment was abandoned in case a fall in the degree of purification should result. The opportunity to develop this point did not again arise, but in any future work, this must be regarded as a point of much importance, controlling as it does the wastage of α TNT and being worthy of full investigation under the existing plant conditions.

---

* At higher temperatures, action upon α TNT increases rapidly ; at lower temperatures, action upon the isomers becomes too slow.

The crystallisers originally in use were cast iron pans, 6 ft. 3 in. diameter × 3 ft. 8 in. deep. The blades, 8 in number, were made of cast steel, serrated on both edges and arranged spirally around the central shaft. The crystalline product has been uniformly good, using this type, but the rate of cooling is slow on account of the small surface area exposed and the fact that the metal wall becomes lagged internally with a coating of TNT which lessens the amount of heat radiated from the walls. The mixing effect at the sulphiting stage is also slow, the first portion of the charge being therefore very thick, and consequently the crystalliser is slow in discharging. With a view to increasing the rate of cooling, several methods were proposed and a few were tried, but, in every case it was found that any attempt to hasten the rate of cooling succeeded only at the expense of the quality of the product, and for these reasons they were abandoned.

The most successful type of crystalliser is illustrated in Figs. 48 and 49. This is made in pitch pine throughout, the dimensions being as indicated. As a result of the increased surface area, this type of crystalliser brings about more rapid cooling than the smaller type referred to above; the figures which follow refer chiefly to the later type of crystalliser.

The outlets of the crystallisers have been recently fitted with treacle valves, and these have given every satisfaction.

Crystallisation process.—The charge of hot water at 90°-100° C. is run into the crystalliser to a fixed dip. A charge of molten TNT averaging 26 cwt. is then run in from the "A" vat to a fixed combined dip. Generally speaking, equal volumes of water and TNT have been found to constitute the best proportion; any excess of water beyond this lengthens the cooling period and serves no useful purpose, whilst a decrease in the quantity of water yields a mixture which in the later stages becomes rather dry, and in this respect exhibits three defects, viz. :—

(1) requires much more power to drive the crystalliser;

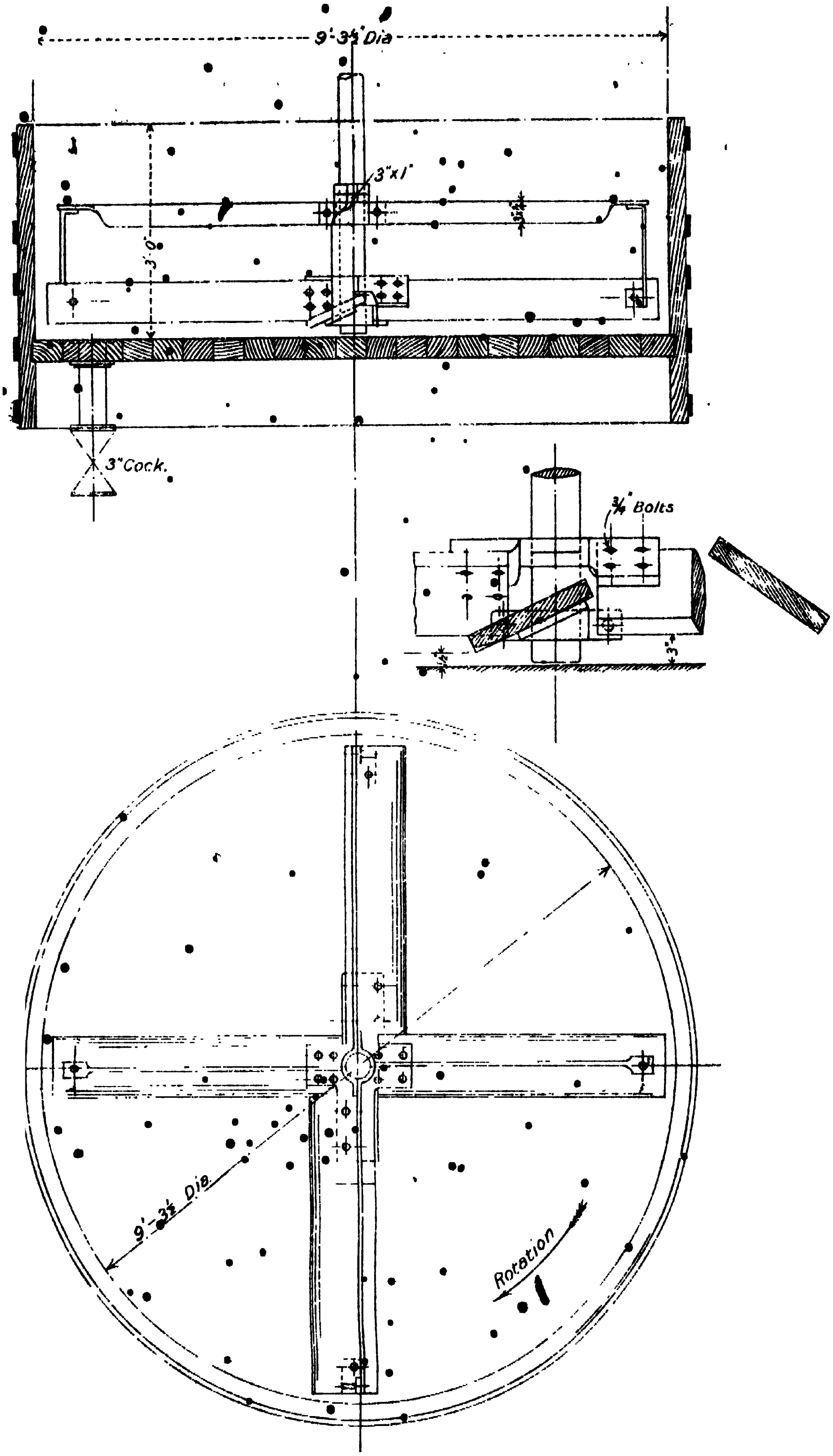
(2) causes attrition of the crystals formed and thus converts the oily eutectic into a paste of TNT dust and eutectic, rendering purification less complete, and incidentally causing trouble in the filters;

(3) increases the wear and tear on the blades.

Employing equal volumes of TNT and water, however, the charge shows little or no supernatant water in the later stages of crystallisation, but remains damp enough to be handled by the blades without very heavy resistance.

The fall in temperature is very rapid in the early stages, the charge dropping from 90° to 75° C. in less than half an hour. As the wet setting point of the TNT is approached, crystallisation commences, and the temperature remains almost constant until crystallisation nears completion, when it again commences to fall. In general, this crystallising period occupies slightly over half an hour, and reduction in temperature after this becomes increasingly slow. Under normal weather conditions, the temperature falls to 50° C. in 4 to 4½ hours from the time of charging; high humidity or specially warm weather increases this period by approximately 25 per cent.

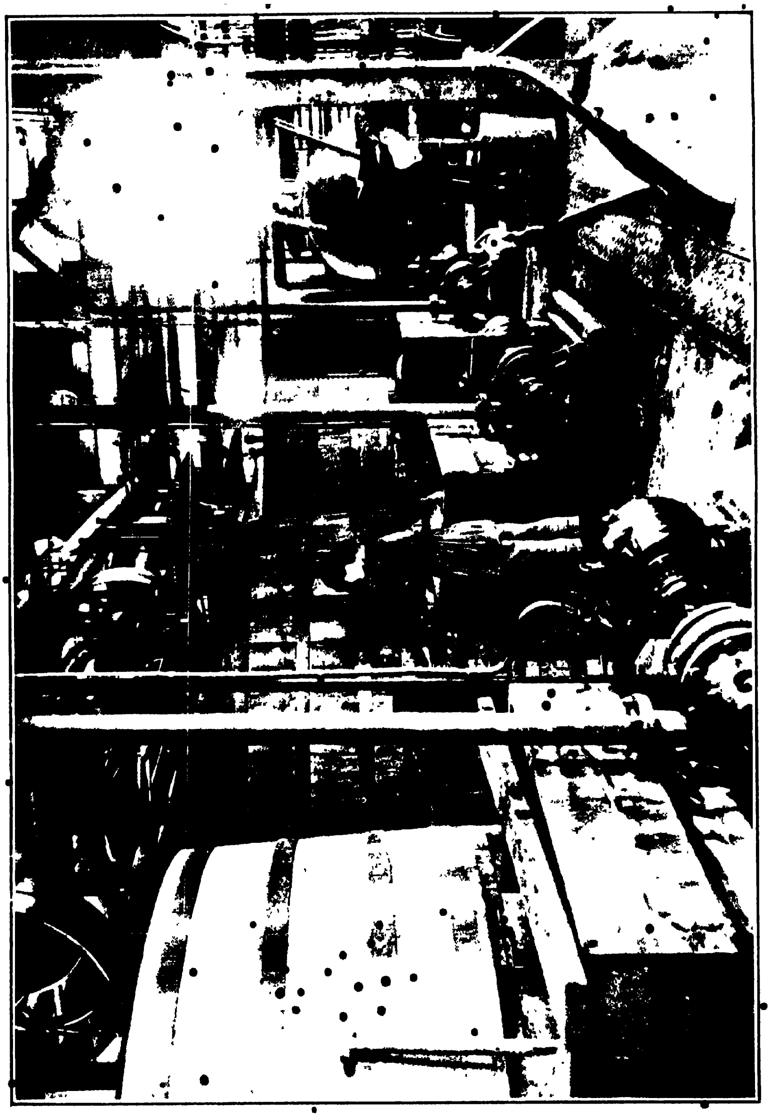
SKETCH OF No. 6 CRYSTALLIZER.



diameter of the vat, and consequent increase in shearing action of the crystals on the blades, resulted in a short life for cast iron or steel blades but for other reasons than this heavy wear the replacement of these blades by something other than iron was rendered imperative. These reasons are discussed under the section dealing with the filter and acid wash vat Wooden blades were found to answer very well; the life is in the neighbourhood of 150 batches, but is somewhat erratic. The blades are four in number, made from single slabs of a suitable hard wood, such as elm. Ash has a tendency to become stringy with wear, and poplar tends to split at the supports. The angle at which the blades are set is a most important factor in this type of crystalliser, an angle of 30° has given the best results. Fig 48 illustrates the blades and the method of attachment. It will be noted that the blade is supported at the end by a bar carried from an overhead straining bar fixed to the shaft. This side bar serves the double purpose of supporting the blade and acting as a side scraper, keeping the sides of the crystalliser free from T.N.T. These scraper bars were at first made of iron but lasted only a few days. A covering of sheet aluminium wrapped around the iron bars gives them a life longer than the blades. The excellent service obtained with aluminium in this case naturally suggests its use for blades in a similar manner. Experiments in this direction, however, were disappointing, the metal cutting badly on the edge meeting the crystals.

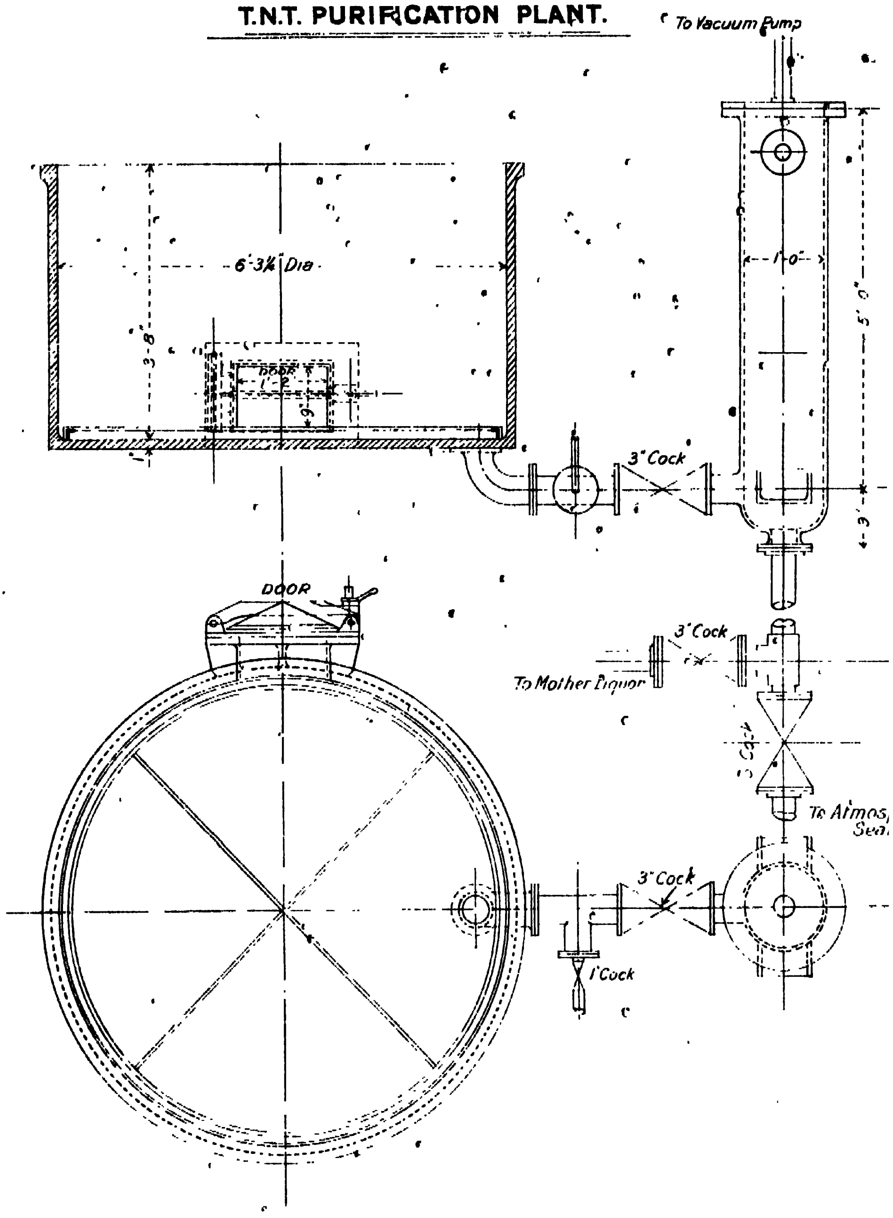
The rapid wear of iron or steel is a matter of some surprise. It was at first thought to be purely a question of attrition, but the experience with aluminium refutes this theory. The suggestion of chemical action upon the metal by the acids remaining in the TNT does not appear to explain the case, since the wear of the central shaft and parts of the blades near the centre is negligible. It is more likely that the action is due to attrition by the crystals indirectly, the clean surface of the metal exposed being superficially oxidised, and the resulting film of oxide being continually swept away. For reasons discussed below, the introduction of iron is specially undesirable. The slight wear in the case of the earlier type of crystalliser blades, however, allows their use to be continued in these crystallisers without any serious deleterious effects in the process. The more recent plant installed in "B" wash-house has also been provided with wooded crystallisers, and these were fitted with wooden stirrer blades which have given good results. The blades are 11 in. wide × 2 in. maximum thickness and are made of ash or elm. Four blades are provided in each crystalliser, these being all on one plane, the lowest edge of each being 2 inches off the bottom, which results in the formation of a layer of TNT 2 inches thick on the bottom of the crystalliser. The surface of this adjusts itself to the path of the blades and the whole of the TNT is properly crystallised. The pitch of the blades is 4 inches in 11 inches along the horizontal, this having proved the most effective arrangement.

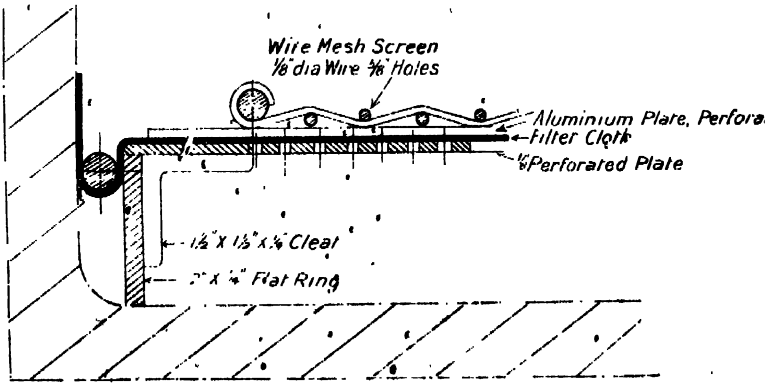
Sludge pumps.—The centrifugal pumps run at 1,400 R.P.M. and each is direct coupled to a motor. A well crystallised batch free from lumps is generally discharged by the pump within eight minutes. Most of the pumps have been of the Dupont type, but any good sand pump is suitable for the purpose. The Dupont pump has given good satisfaction, and the provision of an inspection door enables lumps to be removed and the



SKETCH OF FILTER, ETC.

T.N.T. PURIFICATION PLANT.





impeller examined with a minimum of trouble. Much of the difficulty which was at first experienced with these pumps was due to attempts to pump very thick sludge. Under these conditions, the flow occasionally stopped and the friction generated in the pump fused the mass. It has been found that dilution with mother liquor to yield approximately two parts by weight of TNT crystals to three parts of liquid gives a suitable pumping mixture. In "B" washhouse direct-driven pulsometer sludge-pumps were installed, and these have proved more satisfactory than the centrifugal type referred to above.

The troubles which are met with in running the crystallising plant are mainly mechanical in nature. Although the power consumed by each crystalliser is not large (approximately 4 to 5 H.P.), the slow speed of the average drive necessitates large belt areas for transmission, and the first design did not prove suitable; later both pulleys and belts were enlarged with satisfactory results. Accidental stoppages necessitate at times a large margin of power over that necessary for normal running.

Sludge boxes.—The inefficient mixing in the earlier types of crystallisers led to the discharge of a very thick sludge in the earlier part of the discharge, and the pipe leading from the crystalliser to the pump continually became choked. To obviate this, sludge-boxes (shown in Fig. 50) were installed immediately below the crystalliser discharge cocks, and in these the sludge could be thinned, and temporary blockages of the pump could be more easily dealt with. In the case of the later type of crystalliser, the mixing is excellent and similar reasons for installing sludge boxes do not exist; the boxes were retained, however, to allow for the emptying of the rising main on completion of pumping.

Filter and subsidiary plant.—The function of the filter is to effect the removal by drainage and washing of the soluble sulphonates which have been formed as the result of interaction of the crude TNT with the sulphite liquor. If the sulphonates are not completely removed, the acid treatment which the product undergoes after leaving the filter (discussed later under "B" or acid wash vat) will form free sulphonic acids. The question of the distribution of these sulphonic acids between the water and the TNT is still an open one, and the effect of their presence in TNT is not definitely known. They appear to be very soluble in water, but may be equally soluble in TNT. In addition they appear to have an emulsifying effect on the TNT water mixture, with its consequent process troubles.

The filter and its connections are shown diagrammatically in Fig. 46, and in greater detail in Fig 51. It is a cast iron tank with the filtering medium supported a few inches clear of the bottom. An outlet for drainage is provided, and a door through which the washed crystals are discharged. The drainage of the mother liquor and subsequent washing are expedited by putting the filter under vacuum. The vacuum is obtained by a dry vacuum pump communicating with the filter by an overhead line. The liquid from the filter flows into a Torricellian fall tube provided with a vacuum chamber, which serves as a spray trap, and from the top of which the vacuum line takes off. A storage tank for mother-liquor is situated on the floor below, and the fall pipe branches at this point to supply the tank.

The lower end of the fall pipe, and also the end in the mother liquor tank, discharge into seal pots. The length of the fall tube is about 25 feet, and in practice, a vacuum exceeding 20 inches of mercury cannot be used without danger of liquor rising in the fall pipe and overflowing the vacuum line into the pump. To obviate risk of danger to the pump, a catch-pit has been installed, but the adoption of a wet vacuum pump (as at " B " house) avoids any danger of damage to the pump resulting from the sucking over of liquor. When running to the mother liquor tank in the earlier stages, a vacuum of 4-5 inches of mercury is all that can be used on account of the comparatively small difference in head.

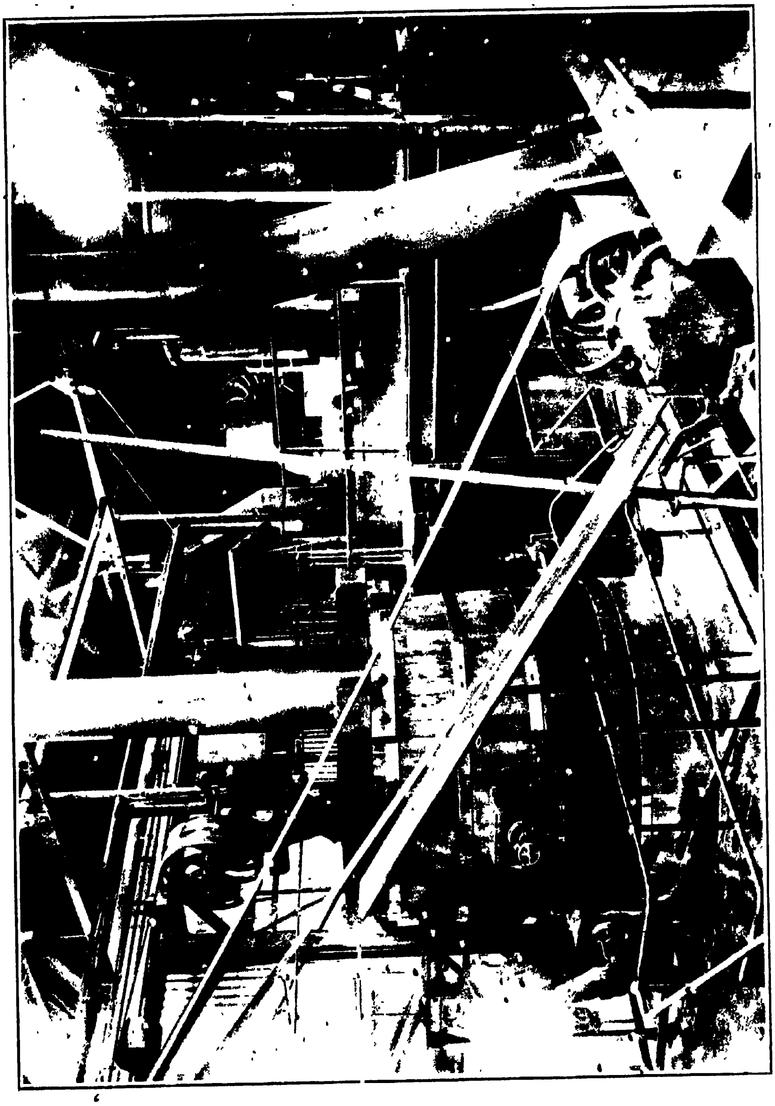
Filtration process.—While the charge is being pumped up from the crystalliser, the outlet cock from the filter and the cock to the mother liquor tank are opened, the down line cock being closed; meanwhile a 5-inch vacuum may be used. On completion of draining, or when the mother liquor tank is full (whichever be first) the cocks at the tee on the down line are reversed and the vacuum increased. Washing is commenced as soon as the surface of the charge is drained. The procedure which has been found to give thorough washing in minimum time is to wash with small successive quantities of water at frequent intervals. In this way displacement of the mother liquor is best effected. In practice, three washes of 4 inches, and later three washes of 2 inches of water suffice. This represents about 200 gallons of wash water. If on emptying the filter the lower levels show traces of red mother liquor, the door is again closed, and the batch given the requisite further washing

On the completion of washing, the batch is drained and operatives then enter the filter and dig the washed crystals into the " B " or acid-wash vat situated immediately below. Several proposals for emptying the filters mechanically have been considered. Emptying by means of a water jet was tried, but did not prove practicable on account of the excessive quantity of water required. Some mechanical stirrer with a slowly descending movement would appear to be a possible method.

The excess of mother liquor is run to the launder and through the settling pits at the recovery plant, where approximately 1 per cent. of the output is recovered and returned to the washhouse.

Two main difficulties were experienced in operating the filters. First, the crystallising in the outlet pipe of TNT from the warm mother liquor. This restricted the delivery of the pipes and consequently increased the tendency for liquor to be carried over the vacuum line, especially in cases of carelessness. The increased number of crystallisers later permitted a longer time-cycle in the crystalliser and consequently yielded a cooler sludge, so that the amount of TNT thrown out in the pipes diminished to negligible proportions.

The second difficulty gave considerably more trouble. It arose from the attrition of the iron crystalliser blades previously referred to. The slight organic acidity remaining in the TNT fed to the crystallisers apparently sufficed to take this iron into solution. On the addition of the alkaline sulphite, however, the soda took the place of the iron, and the latter was thrown out as hydroxide. Generally, this hydroxide remained in colloidal solution, but the aeration in the course of pumping up, and the



washing with a hard water, precipitated the iron hydroxide on the crystals. In this condition, it effectually blocked any ordinary filtering medium, and rendered the passage of water through the crystals themselves a matter of some difficulty. The filter cloths used on the filters rapidly became impassable, frequently after two charges only. These difficulties were met by spreading a layer of granulated TNT, about an inch thick, on top of the filter cloth, and a much coarser variety of cloth could then be employed. The resulting filter bed, heavily charged with iron hydroxide, could then be removed with each charge and as readily replaced by a new one. It may be noted, however, that the iron hydroxide appeared to absorb the mother liquor to some extent, and thus render the removal of the mother liquor an extremely slow proceeding. Further considerations, however, as discussed below, necessitated the elimination of iron. Since then the filters have given little trouble, and the following filtering medium has proved very successful. Resting on the bottom of the filter is a perforated iron base in four quadrants, supported clear of the bottom on 2-inch legs; above this is laid the filter cloth, wedged around the edges of the filter by rope. The material of this cloth is a firm hessian. Above the cloth is laid an aluminium plate, perforated with numerous ¼-inch holes, and finally a coarse ¼-inch mesh wire grid. The purpose of the aluminium plate is to protect the filter cloth; it incidentally prevents the crystals from washing through in the initial stages of pumping in a fresh charge.

A brief mention may here be made of the proposal to dry the crystals at this stage and market the purified TNT in the crystalline form. The physical form of the crystals appears to be ideal, and acid washing (see later) presents no serious difficulties. Drying, in this form, however, would entail more plant and handling than is required for drying the melted product. Further, the drying and sifting of the crystals would be a dusty proceeding and one accompanied by some degree of danger. The appearance of the crystalline product is, moreover, very deceptive. Although it appears very satisfactory, the melt is invariably bad. This is mainly due to minute fibres which have been rubbed off the "A" vats and crystallisers. These fibres cannot be removed by any method of selection or classifying, since in many cases crystals have grown round them. Their presence could perhaps be prevented by the substitution of enamelled vessels, but experience indicates that the latter would have a comparatively brief life. For these reasons it is considered that the present practice of melting, washing, and granulating the crystalline product is preferable. By suitable treatment, a very thin and finely granulated flake can be made, which compares not unfavourably with the crystallised product.

"B" or acid-wash Vat.—The purpose of the next step in the process is to render the TNT acid, and incidentally to melt the product for further treatment. The sulphite used for purification is strongly alkaline, and to ensure that no alkali remains (with its tendency to form dangerous compounds) the TNT is rendered slightly acid.

The "B" vat (see Figs. 46 and 52) is half filled with boiling water, and to this approximately 2 lb. of sulphuric acid (93 per cent) or a corresponding quantity of nitre cake (which has the advantage of freedom from danger in handling) is added; 8 lb. of nitre cake are usually used, this quantity having an acidity which is slightly in excess of the 2 lb. of sulphuric acid mentioned above.

Into this boiling dilute acid the contents of the filter are shovelled, whilst agitation and injection of steam are in progress. The charge when melted is stirred for a few minutes, allowed to settle, and the acid water drained off. The vat is then filled up with boiling water, and the cock connecting it with the "C" or final-wash vat is opened. The TNT is then displaced across to the "C" vat. The working limit leaves two inches of TNT in the "B" vat, to avoid the possibility of any acid water getting across into the "C" vat. The cock is then closed, acid added, and the vat is ready for another charge from the filter.

Considerable trouble has been encountered at this stage of the process. The average life of the wooden stirrer employed does not exceed two months. Trials with cast iron stirrers have proved quite unsatisfactory, the stirrer blade being cut away by the combined action of acid and crystals within a week. The connecting line to the "C" vat has also been a very troublesome feature. Various kinds of straight pipes were tried, but the continual expansion and contraction caused continual leaks at the junctions to the vats. Finally a diagonal line was installed, made entirely in cast iron, with a right-angled bend at each end to allow of fixture to the vat, and this has been quite successful. The joints are made with a thick rubber washer, but most of the expansion appears to be taken up by the bends. A cock is fitted to each end, to enable repairs to the line to be conducted without the necessity of emptying either vat. During the process, however, the cock by the "C" vat is kept permanently open, to avoid any danger of bursting the line between, through change in temperature.

The acid treatment, as outlined above, does not remove iron hydroxide very successfully. The crystals are shovelled into the vat in a moist coherent form, and on striking the boiling water commence to melt as a sticky mass from the outside inwards. During this process part of the iron hydroxide becomes enmeshed in the melting TNT, and being thus surrounded by an oily pellicle of TNT it becomes impervious to the action of the acid water. When this trouble was at its worst, the major portion of the iron hydroxide appeared to remain in the TNT and was thus carried through to the "C" vat. In addition, iron hydroxide is an excellent emulsifying agent, and consequently acid water was carried across to the "C" vat with the TNT, whilst TNT in suspension passed with the wash waters from this vat to the launder. The question rapidly became serious, and, while new blades were being constructed, an endeavour was made to effect the removal of the iron. The means adopted consisted in maintaining the temperature of the dilute acid, into which the crystals are discharged from the filter, below the melting point of TNT. In this way the iron hydroxide is readily attacked and completely removed in the acid water, and subsequent melting does not cause its inclusion in the TNT. The effect of this alteration in procedure was immediate; the finished product became quite bright and emulsification disappeared altogether. This method of treatment had two defects, however, which led to its abandonment as soon as the iron blades were replaced by wood. In the first place the procedure occupied nearly twice the normal time, and thus restricted output, and in the second place, the method proved very severe on the plant

The stirrers wore away rapidly from the attrition of the crystals, and jamming of the drive was of frequent occurrence. Apart from these points, the modification has very great advantages, and if these defects could be overcome, it would probably be advisable to adopt the method, since the present process does not completely remove iron. Even with wooden crystallisers and wooden blades, iron is invariably present in the product in the filter, and a proportion of it passes through to the drier.

"C" or final-wash vat.—The object of this, the last stage in the process prior to drying, is to remove any acid which may have been carried forward from the previous acid-wash.

If the operation in the "B" vat has been correctly carried out, the first wash in this vat is neutral. If such is not the case, however, washing must be continued until the wash water is neutral. The wash water is run off by an upper drain box and returned to the "A" vat, where it serves to wash the crude pellite from the nitration house. The acid free TNT is run off through a lower box direct to the drier (Fig. 52).

With the exception of the replacement of the lead steam pipe by one of silicon-iron, this vat has undergone no alteration. At the time of the iron hydroxide difficulty, however, the separation of the TNT and water in any degree approaching completeness was impossible, since the watery emulsion containing TNT and iron was returned to the "A" vat and thus kept in the system, the effect quickly becoming cumulative.

The "B" vats in the new sulphite washhouse have been raised 12 inches above the "C" vats to facilitate gravitation of molten TNT from one vat to the other.

Fig. 52 shows a portion of the sulphite washhouse in which may be seen the filter (right hand top corner); melt vat (right hand bottom corner); "B" and "C" wash vats; and the run off from the sight-box of the "C" vat to the drier (girl operating valve).

The sulphite process log-sheet is reproduced on the following page.

Preparation of sulphite solution.—The preparation of the sulphite solution has given very little trouble. Waste sulphite from synthetic phenol manufacture has been used throughout. The following analysis gives the average composition of the product supplied:—

	Per Cent.
Na_2SO_3	73.80
Na_2CO_3	1.83
NaOH	0.46
Insoluble	1.54
Water	15.90

The difference consists chiefly of sodium sulphate, and the insoluble matter is mainly iron hydroxide and calcium sulphite. It is important that the alkali content be kept as low as possible.

The solution is prepared in a cylindrical iron vessel, 6 ft. diameter × 3 ft. 6 in. deep, provided with suitable stirring gear. The muddy solution obtained is pumped to a series of settling tanks, from which it is drawn off

I.M. FACTORY, QUEEN'S FERRY.

TNT WASHERS—SULPHITE PURIFICATION.

Wash House........................ 191

A Operator

Batch No........................Large Tip-wagons....................Small Tip-wagons.....................

Set No.........................

Tipped in "A" Vat—Date.................................. Time..........................

No. of Hot Washes.................................. Run to Crystallisers.........................

Cr.

Crystalliser No................................

Dips—Water.................................. Total.............................. TNT...............................

Temps.—Water ....°C. Mixture after starting Stirrer...........................°C

Time—

Charging started at................Charging complete at...............Stirrer started at..............

Temperatures :—

Time.	Temp.	Time.	Temp.	Time.	Temp

Operator

Sulphite Solution—Dip in Tank.................................in. Temp.................................°C

Started adding at.................................. All in at..............................

Mother Liquor added at...........................

Pumping to Filter—Started at................................ Finished at..............................

F Operator

Filter No...........................

Mother Liquor drained off at.............................

Washing finished at.................................. No. of Washes...

Raking to "B" Vat—Started at .... Finished at..................................

B AND C Operator

Acidified at.................................

Running to "C" Vat—Started at.................................. Finished at.............................

No. of Washes in "C" Vat...............................

Separation to Drier—Started at.............................. Finished at..............................

Dip of Drier—Before Separation.................................. After Separation ....

Remarks :

after 24 hours settling and pumped to overhead tanks, whence it gravitates to the wash house. The strength used is approximately a 10 per cent. w/v solution; a higher strength being avoided on account of danger of crystallisation in the pipe-lines during cold weather. Since alkali, more particularly caustic alkali, has a solvent effect upon, and is thus wasteful of TNT without having any marked purifying effect, the presence of this substance in the sulphite is undesirable. Partial acidification of the solution with nitre cake was tried, and analyses indicated that the alkali was sensibly reduced with little apparent action on the sulphite. This work was in progress when operations ceased; but it is deserving of further investigation. The figures obtained were as follows :—

	Sulphite solution before adding nitrate cake. Per cent.	Sulphite solution in which nitre cake in the proportion of 56 lb. to 700 lb. of crude sulphite has been added. Per cent.
Na_2SO_3	11.40	11.20
Na_2CO_3	0.40	0.18
NaOH	0.04	0.014

Rise in setting point by sulphite washing.—The rise in setting point by sulphite washing has maintained a very constant average of almost 2.2° C. A report on this method by the Research Department, Woolwich, indicates the rise of setting point of TNT (containing 4 per cent. of β and γ isomers) to be 2.4° C. The figure quoted above may, therefore, be considered very satisfactory for plant practice.

Each batch of crude pellite is tested before tipping in the "A" vat. The lowest limit allowed for Grade II. is a wet setting point of 73.7° C., the lower limt for Grade I. being 74.2° C. These wet setting point values carry a correction of 3.7° to dry setting point, and with the further addition of 2.2° for sulphiting, yield the following figures :—

Grade II.	° C.	Grade I.	° C.
W.S.P.	73.7	W.S.P.	74.2
W.S.P. to D.S.P. ...	3.7	W.S.P. to D.S.P. ...	3.7
Sulphiting rise ...	2.2	Sulphiting rise ..	2.2
	79.6		80.1

Loss of TNT in sulphite washing.—Several attempts have been made to determine the loss in purification, but the conditions obtaining at Queen's Ferry did not, for a variety of reasons, permit of accurate determinations. The β and γ isomers, which average 4½ per cent. on the crude material, are almost completely removed, and, in addition, under the intensive working of the plant and consequent occasional sulphiting in the region of 50°

to 60° C., an amount of TNT probably in the neighbourhood of 3½ per cent. has been removed. This would give a total loss of 8 per cent., which figure could be appreciably improved upon by sulphiting between 40° and 45° C.

DRYING.

General.—The TNT running into the dryers from the washhouse possesses the following characteristics:—

(a) It contains a small amount of sediment consisting of lead sludge, grit, so-called "transformation product," and wood fibre from the wash vats.

(b) It contains 1.1 per cent, of water in solution at 85° C.

(c) In addition to the dissolved water, it is mixed with as much as 0.5 per cent. of water in the form of an emulsion; this soon breaks down at temperatures exceeding 100° C.

Sediment.—The bulk of the heavy matter settles out in the first drier. This necessitates the cleaning out of the driers once every four months, when there is normally about 2 inches of sediment in the top drier, 1 inch in the middle drier, and ½ inch in the bottom drier. The detonating point of this deposit is somewhat lower than that of crude TNT, varying from 280° to 300° C., the detonating point for normal TNT being 313° C.

The continuous washing plants affect the drier deposits in the following ways:—

(1) In the house in which the first (wooden) continuous washer was installed, the drier deposits were much increased on account of the large amount of wood fibre from the washer and sludge from the lead steam pipes.

(2) In the driers attached to the cast iron continuous washer, the amount of sediment is much smaller.

The lighter matter, *e.g.*, wood fibre, passes down through the driers and, if not removed, passes over the granulator roll together with the TNT. This is certainly small in amount, and can only be removed by filtering.

The original drying scheme provided for the filtering of the TNT after it had passed through the first two driers. This was much criticised at the time, but experience in the sulphite process has since confirmed the suitability of this arrangement. It proved quite impossible to filter TNT in its wet emulsified state, the fine gauze filter (60 mesh) becoming rapidly choked, but little difficulty was experienced in filtering the TNT after it had passed through the driers. The most suitable stage for filtration is before the TNT is cooled for granulation, a 60-mesh phosphor-bronze trough-shaped sieve being quite satisfactory for this purpose.

Emulsion of TNT and water.—The greater part of the water combined with the TNT in the form of an emulsion separates out and evaporates in the first drier

Water dissolved in TNT.—The water in solution can be removed by keeping the TNT for a sufficiently long time at any temperature above 100° C. In practice, a period of 2 hours at 110° C. is found sufficient.

DISCONTINUOUS DRYING.

Discontinuous driers.—A complete discontinuous process drying unit comprises two sets of three driers each.

Each drier consists of a rectangular wrought iron tank, 10 ft. × 6 ft. × 2 ft. 3 in. deep, with a slope of 2 inches towards the outlet, and a working capacity of 4½ tons of TNT. The latter flows down by gravity from drier to drier through steam-jacketed wrought iron pipes fitted with cast iron steam-jacketed cocks. Each drier is fitted with four 1-inch closed wrought iron steam coils, supported on angle irons one inch from the bottom, and supplied with low-pressure steam at 30 lb. per square inch. The total length of coil is approximately 80 feet. The exhaust steam from the driers is utilised to heat the dish under the granulator roll. The steam required for heating the jacketed pipes and cocks is provided by means of a by-pass on the main drier system. The fumes given off during drying were at one time drawn off by means of 4-inch pottery pipes attached to the aluminium covers of the driers, and connected to the fume exhauster of the granulators. These were later cut out in order to increase the draught on the granulators, and 12-inch vertical sheet iron ventilating shafts were fitted to the tops of the driers which pass through the roof and discharge into the atmosphere.

Each dryer is connected to a safety device whereby its contents may be drowned with water from the fire main in case a fire occurs in the building. This emergency system is brought into operation by means of valves situated at various places easy of access outside the building.

Discontinuous drying process.—The final wash vat delivers the washed TNT to the first drier of each set, which serves as a settling or storage tank for the remainder of the drying system. This is heated by steam coils and kept at a temperature approaching 100° C. In this tank a small separation of water carried mechanically with the TNT takes place, but in the interval between delivery of successive charges the water is completely boiled off. If, however, any emulsification is taking place in the washers, a much larger quantity of water is brought down with the TNT and tends to accumulate in the drier. For this reason, the appearance of the TNT in this drier tank reflects very markedly the working conditions of the process. Besides the preliminary drying which this tank affords, it serves to settle out grit, slime, and general débris from the TNT. Since this layer of débris is very difficult to remove, however, several attempts have been made to filter or even to strain the TNT between the final wash vat and this drier tank. Such attempts have all been unsuccessful on account of the semi-emulsified nature of the product. It has only been found possible to filter the product after settling and partially drying; for this reason filters are interposed between the feed tank and the driers.

Filter.—The filters are shallow gauze baskets, one of which is set inside the top end of each drier under the delivery pipe from the feed tank. They are of 80-mesh phosphor-bronze wire, steel or tinned steel being useless on account of rusting. They are designed as simply as possible, to permit of rapid cleaning, which is effected by turning them upside down and blowing steam through the gauze. Normally, the residue is a mass of fine wood fibre which must be removed once every shift. If any appreciable amount of iron hydroxide is present in the TNT, however, the filters become inoperative very rapidly.

TNT enters the first drier (or settling tank) at approximately 85° C., and the temperature is here raised as nearly as possible to 103° C. The main part of the drying takes place in the second drier, in which the TNT remains undisturbed for a period of 2 hours at a temperature of 105°-115° C. The third drier serves merely as a cooling vessel, in which the temperature of the TNT is allowed to fall to 85° C., this temperature being the most suitable one at which to granulate the product.

During operation, a steady flow of TNT is allowed to pass from the third drier into the steam-jacketed tray beneath the granulator roll. When this drier is empty it receives the full charge from the second drier, the contents of which have been kept undisturbed for a period of not less than two hours. The second drier, when empty, receives the full charge from the first drier.

Cleaning of driers.—The following procedure is adopted for cleaning the driers. The driers are emptied as completely as possible of TNT, and allowed to cool. The covers are then removed and the sediment chipped off with chisels and mallets of any suitable hard wood, the operation being completed by a jet of steam. The deposit when removed is burnt.

CONTINUOUS DRYING.

Continuous drying plant and process.—Continuous drying plants (see page 15) have been installed in 3 of the 5 TNT units. In the continuous process, TNT flows from the first drier or storage tank, referred to above, in the form of a thin film 1/20 inch in thickness, along a wide shallow iron channel which is heated by means of a steam-jacket into which steam is passed at a pressure of 30 lb. per square inch. The TNT leaving the drier flows into a cooling trough whence it passes direct to the granulator (Fig. 46). Thus each of the two continuous drier sets dispenses with two of the large driers used in the discontinuous process, and the quantity of TNT in process is therefore greatly reduced.

The entire plant for each unit consists of 2 drier sets, each comprising feed tank (the original first drier in the discontinuous process), 2 steam-jacketed continuous driers, and a cooling channel.

Each drier consists of a hot plate, 12 ft. long × 2 ft. wide, having a fall in its length of 1 inch; margin plates are bolted to the edge of the plate to prevent TNT flowing over the sides. The flow of TNT from the feed tank is regulated by means of a regulus metal screw-down plug-valve, with a rubber inset and operated by a spindle which extends through the cover. From the first drier the TNT flows along a channel iron trough to the second drier, whence it passes through a series of cooling channels to the granulator.

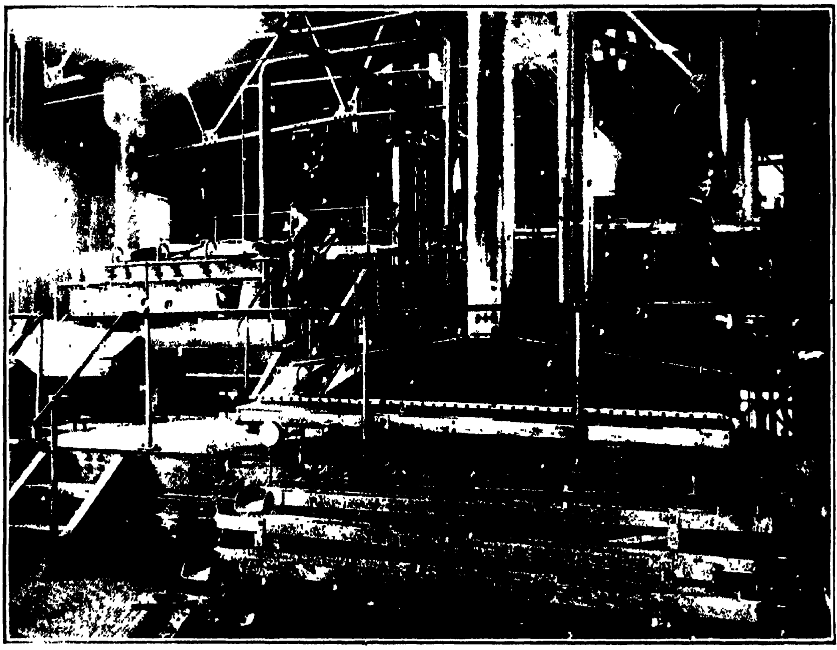


FIG. 54.

To face p. 103.

FIG. 55.

To face p. 103.

The temperatures of the TNT and its volatile content (V.M.) at the various stages of the process are given in Fig. 53. The general arrangement of the building is indicated in Fig. 54, which shows girls operating the steam valves on the feed tanks, and also the continuous driers and cooling channel to the granulators.

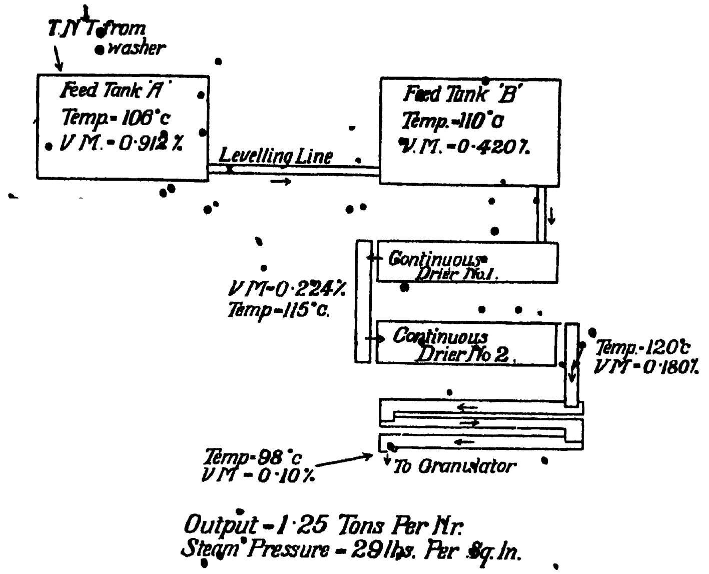


FIG. 53.—CONTINUOUS DRYING PLANT TEST RUN.

GRANULATING, PACKING AND STORAGE.

General.—The plant for this purpose consists in each unit of two granulators, one weighing machine, a system of conveyers, a screwing machine, and trolleys for conveying the packed boxes of TNT to the magazine.

GRANULATION.

The granulation of the dried TNT is effected by gunmetal drums (Fig. 55) internally cooled by running water, which rotate in a shallow tray containing the molten TNT. A layer of TNT is picked up, which after solidifying on the drum is removed by a knife-edge. The granulation of sulphited TNT is a very dusty proceeding compared with that of the less purified product, on account of the removal of the low setting point impurities. At "A" washhouse new dust flues have been erected, and a device for the removal of the dust in a wet state was in contemplation when the plant was shut down.

Granulator.—The granulator consists of a hollow gunmetal drum, 2 ft. diameter × 3 ft. long, the thickness of the shell when new being approximately ¾ inch. This is revolved at a rate of 12 R.P.M. for the lower grade of TNT; for the production of the highest grade of product the speed is reduced to about 4½ R.P.M. The drum is supported on trunnion bearings, through one of which the cooling water enters by means of a ½-inch pipe which is directed to the bottom of the drum. The warm water overflows through the trunnion bearing at the other end, so that the drum is always half full of water. The drum is arranged so that it dips into a steam-jacketed bath, which consists of a cast iron base with a brass pan ⅜ inch thick. The pan is heated by the exhaust steam from the TNT storage tank feeding the continuous driers. The granulating knife consists of a gun-metal blade, 5 in. deep × ⅜ in. thick, one edge of which is brought to a chisel point; this edge rests against the roll for its entire length. The blade is attached to a steel back which is held in position by two brackets fixed to the cast iron "A" frames supporting the trunnion bearings. The whole is encased in a light sheet iron covering provided at the top with a fume pipe and below with a chute to convey the TNT to the weighing tins. The general arrangement is shown in Fig. 46.

Fume system.—The original equipment consisted of a 4-inch fume take-off from each granulator, leading to a 6-inch fume-main which opened into a 24-inch earthenware main leading to two 25-inch fans direct-driven by electric motors and operated alternately.

PACKING.

Weighing arrangements.—The weighing machines are of the plus or minus recording type. The TNT is weighed in liner-bags in tins of standard weight (Fig. 56).

Conveyers.—The liners when filled are tied up, tipped out of the tins, and placed in wooden boxes. The supply of empty boxes to the weighing table and of full boxes to the screwing machine is carried out by means of mechanical conveyers (Figs. 56 and 57).

Screwing machine.—The lids of the boxes are screwed on by means of an automatic screwing machine (Fig. 57), one of which is installed in each packing house. The boxes are then taken, 12 at a time, on flat trolleys along a 24-inch gauge tramway to the magazine.

STORAGE.

Magazine.—Each magazine is 50 ft. × 32 ft. × 8 ft. high from floor to springing of roof (Fig. 58). All lighting of the magazine is external, electric globes being fitted in special water-tight boxes outside the skylights in the roof.

RECOVERY PLANT.

Treatment of contaminated TNT.—A varying quantity of contaminated TNT, which results from the processes already described, is collected from various sources for recovery of the TNT and other nitro bodies contained in it. The treatment which such material undergoes varies largely with its source and also with its purity. Thus, TNT resulting from spills in the washing houses, or from the wash-water cooling launders, is conveyed direct to the washers, where it is mixed with a charge of pellite from the nitration

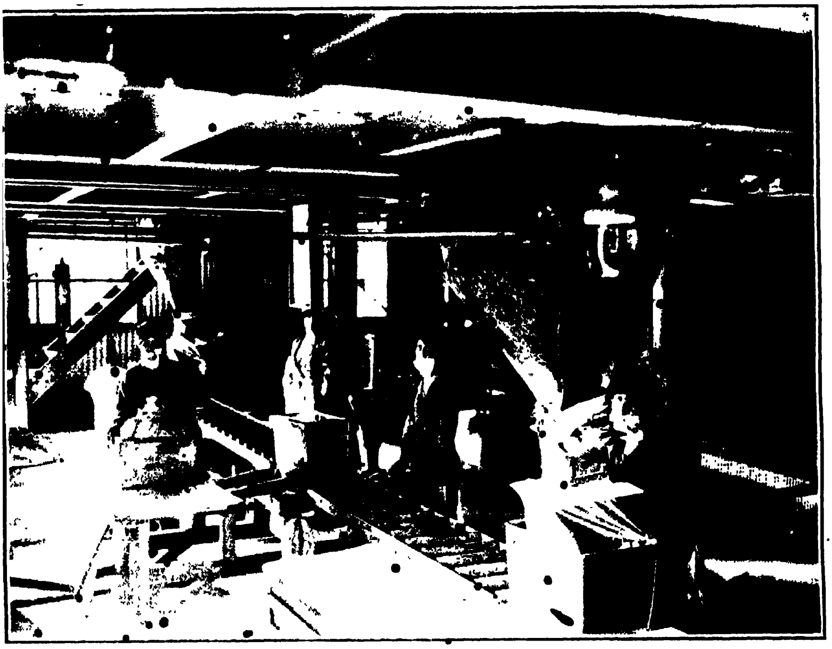


FIG. 56.

[To face p. 104.

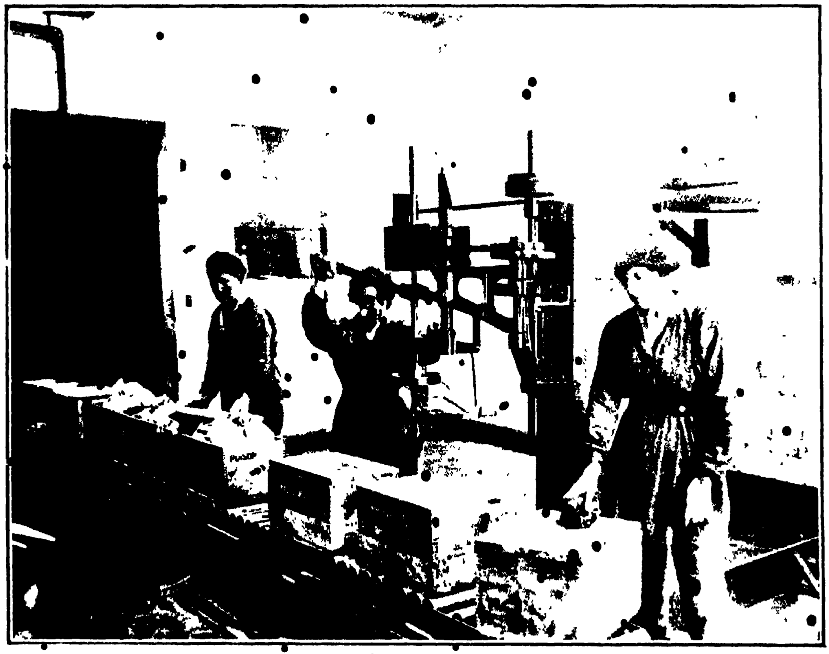


FIG. 57.

[To face p. 104.

FIG. 58.

To face p. 102.]

house and re-washed. The deposit which accumulates in the far end of the cooling launders is removed into boxes and allowed to drain before being carried to the recovery plant where it is mixed with dirty TNT from floor sweepings, effluent drain catch-pits, bag shaking plant, drips from cocks, etc.

Solid nitro-bodies other than TNT are conveyed to the recovery plant from various sources on the Acids Section. The most important points at which solid nitro-body is deposited are the spent acid storage tanks feeding the denitrators, the fans at the Gaillard towers, and the Cottrell fume precipitator.

The contaminated material from the TNT and Acids Sections are melted separately in lead vats fitted with perforated false bottoms, the molten nitro-body in both cases being drawn off into tapered iron moulds of mean diameter 23 inches and approximately 16 inches deep (obtained by cutting disused steel drums in two at the largest diameter), in which it is allowed to cool. Cooling occupies about 3 days for TNT and considerably longer for nitro-bodies of lower setting point. The solidified contents of the moulds form in two layers. The top layer, which is 2-4 inches thick, consists for the most part of dirt, etc., previously mixed with the nitro-body. This layer is scraped off and removed in boxes to the burning ground. The moulds are then inverted and the cast nitro-body removed and broken up into lumps. The product resulting from this treatment is of high setting point, and if of good colour it is loaded into tip-wagons and dispatched to the nearest washing house and re-introduced into the washers.

Nitro-body of low setting point is submitted to a modified nitration process. The acid charge consists of a mixture of mixed acid (as used for the trinitration) and sulphuric acid of 94 per cent. strength, the exact proportions of the two depending upon the gravity of the nitro-body, the general practice being to use equal parts of each. The charge of nitro-body is 12 cwt. to about 4 tons of combined acids. Nitration is effected in a vessel similar in all respects to a trinitrator (page 49), which is fitted with a 2-way steam-jacketed plug-cock situated 2 ft. 9 in. from the top of the vessel. The temperature is raised 5° every 10 minutes to 100° C. When the desired setting point is reached, the charge is cooled and diluted as in the ordinary trinitration process. The stirrer is then stopped, and the charge left standing in the vessel for three-quarters of an hour to separate.

The TNT is then separated off through the side outlet, and the spent acid is run into a separation vessel in which any TNT which runs off with the acid rises to the surface and is skimmed off, thus enabling the acid to be pumped, fairly free from nitro-body, to the spent acid storage tanks.

At one time it was found advisable to treat the recovered TNT from the melt vats with 96 per cent. sulphuric acid in the nitrating vessel at the recovery plant. The product, which was otherwise very dark, was by this means obtained in a clear condition and of good colour. With the introduction of the casting method described above, however, the cast blocks of TNT were sufficiently pure to be introduced into the washers in the washing house without further treatment.

Treatment of material contaminated with TNT.—Lead scrap from the TNT section is treated as follows. Some of the lead cooling coils from the nitrators or detoluators are cut into lengths and washed with boiling water. They are then utilised as steam-heating pipes for wash tubs in the washing house and elsewhere. The rest of the coils, together with all other forms of scrap lead, are cut into convenient lengths and removed to the lead washing plant. The lead is here loaded into an iron cradle attached to an overhead run-way, and the cradle is dropped into boiling water contained in a large iron vessel. The lead is then removed and trolleyed to the burning ground where it is melted and cast into pigs.

Iron, aluminium, brass, etc., is made to undergo a similar washing treatment before being re-used or passed as scrap.

EFFLUENT DISPOSAL.

The effluent drain, into which are fed the wash-waters from the nitration and washing houses, is constructed of 12-inch split pottery pipes which are bedded in brickwork, molten sulphur being run into the spaces between the pottery sections in order to produce an acid resisting joint. The drain has an average fall of 2 inches in 100 feet length on the TNT section, and is broken by two catch pits alongside the recovery plant (Fig. 59). These pits hold up any TNT which finds its way into the drain, and their contents are removed at intervals to undergo purification in the recovery plant. The wash-waters, after passing through the catch pits, flow into one of three neutralising vats, 20 ft. diameter × 6 ft. deep, of 8,000 gallons capacity. The contents of the vats are agitated by means of a cast iron propeller running at 200 R.P.M., which causes circulation at the rate of 2,000 gallons per minute, so that the total contents of the vats are turned over every four minutes. Whilst agitation is in progress, milk of lime is run in from a lime mixer until the contents of the vat are no longer acid. The neutral effluent is then discharged by means of 4-inch centrifugal pumps into one of three

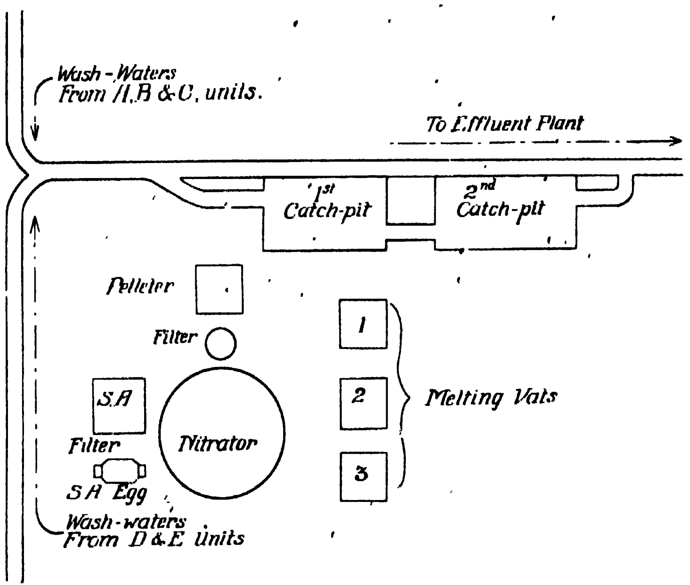


Fig. 59.

precipitation tanks, 60 ft. long × 19 ft. wide × 7 ft. deep, each of 40,000 gallons capacity. The effluent, after settling, passes through two sand filters and flows into a sump whence it is drawn by one of two plunger-pumps which discharges it at the rate of 200 gallons per minute through a 6-inch pipe-line to the River Mersey, a distance of several miles. The deposit which accumulates in the settling tanks is pumped at intervals by means of a sludge pump into a sump adjoining the effluent plant.

STATISTICAL RECORDS.

It is not intended to give a detailed account here of the system of statistical control in operation at the factory, as this has already been set out at considerable length in two previous publications from H.M. Stationery Office,* in the second of which is given a complete set of the technical and statistical records used at Queen's Ferry.

A brief reference may be made, however, to the utilisation of such records in analysing various phases of the process. Thus, a series of sulphur, carbon, and nitrogen balances have been systematically drawn up with a view to allocating accurately and with precision the various sources of loss of materials. As an example of this, there is reproduced on the following page a table showing the distribution of nitrogen in process for the period 6 a.m., August 3rd, 1918, to 6 a.m., August 31st, of the same year, and the same figures are set out in graphical form in Fig. 60. It will be at once evident that the preparation of tables and charts of this kind calls for a very careful system of plant control, accurate stocktaking, and numerous chemical tests, if such records are to be of any real value. In carrying out work of this kind many obscure sources of loss are revealed, and it is in the following up and investigation of these losses that very real benefit to the general efficiency of the process is obtained.

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* "Second Report on Cost and Efficiencies for H.M. Factories Controlled by Factories Branch, Department of Explosives Supply."
"Report on the Statistical Work of the Factories Branch."

H.M. FACTORY, QUEEN'S FERRY.

DISTRIBUTION OF NITROGEN IN PROCESS.

Period:—6 a.m., August 3rd, to 6 a.m., August 31st, 1918.

Basis:— 809.42 tons toluene
1166.76 tons MNT.
1734.368 tons TNT.

			APPROXIMATE DISTRIBUTION OF LOSSES.			
Charged to Plant.	Recovered from Plant.	Loss of Nitrogen.	Handling and on Bags.	In exit gases.	HNO₃ in Nitre Cake.	In Effluent.
2503.88 tons crude sodium nitrate. 51.10 tons provisional loss. 2554.98 Equivalent to:— 1815.33 tons .... HNO_3 402.96 tons .... N_2	HNO_3 .... 1698.79 tons. Equivalent to:— N_2 ... . 377.51 tons. Efficiency .. . 93.69%	HNO_3 .... 114.54 tons. Equivalent to:— N_2 ... . 25.45 tons. % Loss... .. .. 6.31%	Provisional Loss..... 3.00% Less Recovery at Bag Washer 1.00% 2.00%	3.24%	Nil.	1.07%

TRINITRATION STAGE.

Charged to Plant.		Passed Forward.	Recovered.	Total.	Total.	APPROXIMATE DISTRIBUTION OF LOSSES.			
As HNO_3.	As MNT.	T.U.A. passed to Denitrators.	As TNT.	Recovery.	Loss.	Oxidation and Secondary reaction.	Fumes.	To Washing as O.U.A.	Unaccounted for.
1482.38 tons ......HNO_3 Equivalent to:— 329.42 tons Nitrogen.	1166.76 tons ... MNT. Equivalent to:— HNO_3 .. .. 536.54 tons. N_2 . .. 129.23 tons.	HNO_3 Used in MNT. Mixes Nil. Used in TNT. Mixes .. Nil. Sent direct to Denitrators. 371.44 371.44 Equivalent to:— N_2 ... . 82.54 tons.	1734.368 tons TNT. Equivalent to — HNO_3 1444.05 tons. N_2 . . 320.90 tons.	N_2 As MNT . 320.90 As Spent Acid 82.54 403.44	Total Used .. 448.65 Total Recovr'd 403.44 Loss 45.21 10.08%				

MONONITRATION STAGE.

Charged to Plant.		Passed Forward. (Spent.)	Recovered.	Total.	Total.	APPROXIMATE DISTRIBUTION OF LOSSES.			
As HNO_3.	As Toluene.	To Denitrators.	As MNT.	Recovery.	Loss.	Oxidation and Secondary reaction.	Fumes.	To Washing as O.U.A.	Unaccounted for.
570.02 tons ..... HNO_3. Equivalent to:— 126.67 tons ...... N_2.	837.81 tons. Toluene. Equivalent to:— HNO_3 .... Nil. N_2... .... Nil.	Tons. Equivalent HNO_3 in Spent Acid .. . . 13.84 Equivalent to: N_2 ... ... 3.08	1166.76 tons . MNT. Equivalent to:— HNO_3 . 536.54 tons. N_2 . .... 119.23 tons.	As MNT.... ... 119.23 As Spent Acid. 3.08 122.31	Total Used .. 126.67 Total Recovr'd 122.31 Loss 4.36 3.44%	Nil.	0.035%	1.52%	1.885%

DENITRATION PLANT. 91.78% EFFICIENCY.

			APPROXIMATE DISTRIBUTION OF LOSSES.			
Charged to Plant.	Recovery.	Loss of Nitrogen.	In Denitrated Acid.	In exit as Nitrogen Oxides.	Leaks.	Losses in Blowing, etc.
Spent containing: Equivalent HNO_3........ ... 385.28 tons. Equivalent N_2 .... 85.62 tons.	353.61 tons .... HNO_3 78.58 tons .... N_2 Efficiency 91.78%	Nitrogen Charged... . 85.62 tons. Nitrogen Recovered 78.58 tons. Loss ... . 7.04 tons. Loss 8.22%	3.5 tons Nitrogen. = 4.09%	1.30%	2.75%	Nil.

Total Loss of Nitrogen in the production of 1734.368 tons TNT from 837.81 tons Toluene.

	Tons.	%
At Retorts	25.45	6.31
In TNT Nitration	45.21	11.22
In MNT Nitration	4.36	1.08
In Denitration.............	7.04	1.75
	82.06	20.36

	%
Recovered as TNT	79.64
Loss...	20.36
	100.00

SECTION 3.

PLANT AND PROCESS FOR THE MANUFACTURE OF 2 : 4-DINITROTOLUENE.

General.—The manufacture of 2 : 4-dinitrotoluene, for use in the preparation of nitrocellulose powder, is carried out at Queen's Ferry by treating crude mononitrotoluene with a mixed acid of the same composition as that used for the mononitration (see page 34).

Nitrator and subsidiary plant details.—The acid is fed from a storage tank into two measuring tanks placed outside the nitrating house, each 5 ft. diameter × 6 ft. deep. Each measuring pot is provided with a glass float (see page 31), calibrated in $\frac{1}{4}$ inches. A nitrating charge of 2.8 tons (17 mols. HNO_3) corresponds to 145 marks on the float, and is run direct from the measuring tank to the nitrator (Fig. 3). The latter, which is similar in all respects to that described on page 31, contains two sets of $1\frac{1}{2}$-inch lead cooling coils, supported by cast iron standards (Fig. 4). One of the coils may be used alternatively for steam or water. The cooling water which passes through the coils is drawn from two 9 ft. × 30 ft. steel tanks, one at an elevation of 26 feet to serve the nitrators, and one at an elevation of 19 feet to serve the detoluators; a constant head is obtained by means of a float-valve operating inside the tank. The water is drawn through the nitrator and detoluator coils by two steam pumps; a horizontal steam engine for driving the shafting, and an air compressor (for delivering 400 cu. ft. of free air per minute at a pressure of 45 lb. per square inch) are also provided.

The sight-box through which the charge is run from the nitrator is similar to that shown in Fig. 36. It consists of a rectangular cast iron open box, 12 in. × 8 in. × 12 in., and one inch in thickness, provided with a 2 inch inlet and outlet. The sight-box is bolted direct to the nitrator, and the same bolts are used to keep in position a renewable cast iron valve seat. The plug is of regulus metal, and is attached to a regulus spindle with a $\frac{5}{8}$-inch iron core. The operating gear for the plug is made up from a scrap 3-inch regulus valve.

Nitration process.—The nitration stages are comparable to those already described for TNT manufacture, and consist of a dinitration stage and a detoluation stage. In the latter the acid resulting from the dinitration is washed with crude mononitrotoluene in order to produce a clear acid, *i.e.*, as free as possible from solid nitro-bodies, suitable for delivery over a long distance to the Acids Section. The nitro-body ("acid-MNT") is then used in the dinitration.

When the acid-MNT has been blown up from the detoluator into the nitrator, the stirrer is started and the mixed acid run in at such a rate that the temperature is below 60° C. when all the acid has been admitted; this operation occupies about $1\frac{1}{4}$ hours. The charge is now warmed up to 90° C. by passing steam through the steam coil, and requires 1 hour to rise to this

H.M. FACTORY. QUEEN'S FERRY.

DNT Section.

DINITRATION RECORD.

Pan........................ Batch........................ Date........................

MNT Charge..lb. (S.G........................) from Detoluator

Acid Charge ....lb. Acid Flow started at ....

„ „ finished at........................

Temp. reached 90° C. at........................ Stirrer stopped at........................

Separation started........................ Finished........................

Total time of Operation........................

	Time Period.	Times.	Temp. due.	Temp. observed.	Stand of Float Reading.	Acid Marks.	Remarks.
Running in acid up to 60° C. in 1 hr. 15 min.	Hr. Min.						
	0 0						
	15		35				
	30		45				
	45		50				
	1 0		55				
Heating period 60-90° C. in 1 hour.	15		60				
	30		70				
	45		80				
	2 0		85				
	15		90				S
Heating period 90° C.	30		90				
	45		90				S
	3 0						Setting Points.
	15						Time. Result.
	30						
	45						
	4 0						
	15						
	30						
	5 0						

Signature..

temperature, at which it is kept until the wet setting point of 51°-52° C. is reached (about ¾ hour). When the charge has reached 90° C., and afterwards at intervals of ¼ hour, samples are taken for determining the wet setting point. The final wet setting point should be approximately 51°-52° C., and this should ensure a dry setting point of 54°-55° C., corresponding eventually to a dry setting point of 66°-68° C. after the product has been dried and centrifuged.

The charge is then cooled to 70° C.; the stirrer stopped, and the charge allowed to stand for at least ¾ hour. After the charge has settled the specified time, the once used acid is separated from the molten dinitrotoluenes and run through a 2-inch pipe to the detoluator, the separation being controlled by means of the small sight-box described above. The once used acid has the following composition:—

	Per Cent.
H_2SO_4	71.5
HNO_3	1.5
HNO_2	1.0

Detoluator.—The detoluator is similar in all respects to the nitrator.

Detoluation process.—Crude MNT is pumped from the large storage tanks at the mononitration plant to a storage tank, holding 60 tons, situated on the DNT plant. It is run by gravity from this tank into two measuring vessels fitted with gauge glasses, 18 inches on which correspond to the detoluator charge of 1.15 tons. The MNT (sp. gr. 1.17 at 15° C.) is run from these tanks into the detoluators through a 2-inch pipe; the stirrer is started, and the once used acid from the nitrator is run into the MNT charge at a temperature of about 65° C. This operation occupies about ¼ hour. The combined charge is then cooled to 25° C., allowed to stand for about an hour, and then separated.

The average composition of the spent acid is:—

	Per Cent.
H_2SO_4	72.0
HNO_3	Trace
HNO_2	1.3

The spent acid is run through a sight-box, similar to that described above, into a lead-lined blow-cask, and blown to a spent acid storage tank in the mononitration plant, from which it gravitates to the storage tanks at the denitration plant on the Acids Section. The acid-MNT is then blown up to the nitrator for the dinitration stage.

Pelleting and washing.—After the once used acid has been separated and run to the detoluator, the molten crude DNT is pelleted in much the same manner as that carried out for TNT (see page 76), in a series of wash vats arranged in cascade.

Pelleter.—The pelleter (Fig. 61) consists of a square wooden vat 10 ft. 3 in. long × 9 ft. 9 in. wide, of average depth 2 ft. 10½ in., and having

H.M. FACTORY. **QUEEN'S FERRY.**

DNT Section.

DETOLUATION RECORD.

Pan.......................... Batch................................ Date..................................

M.N.T. Charge..............................lb. O.U.A. Started at..

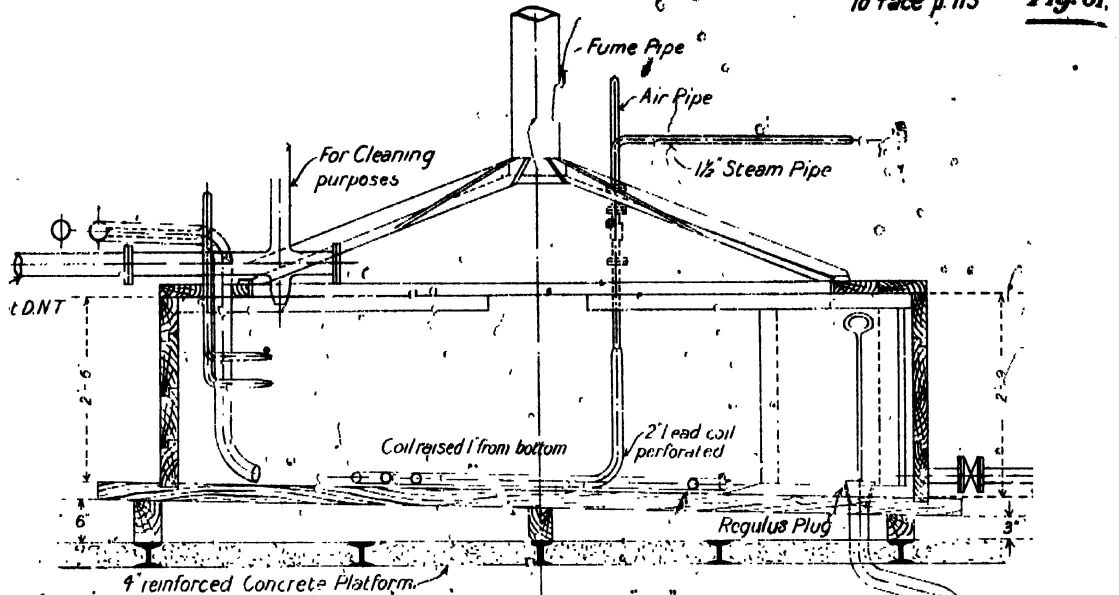
Stirrer stopped at...................................... Separation started at.......................................

Finished .... Total time of operation ....

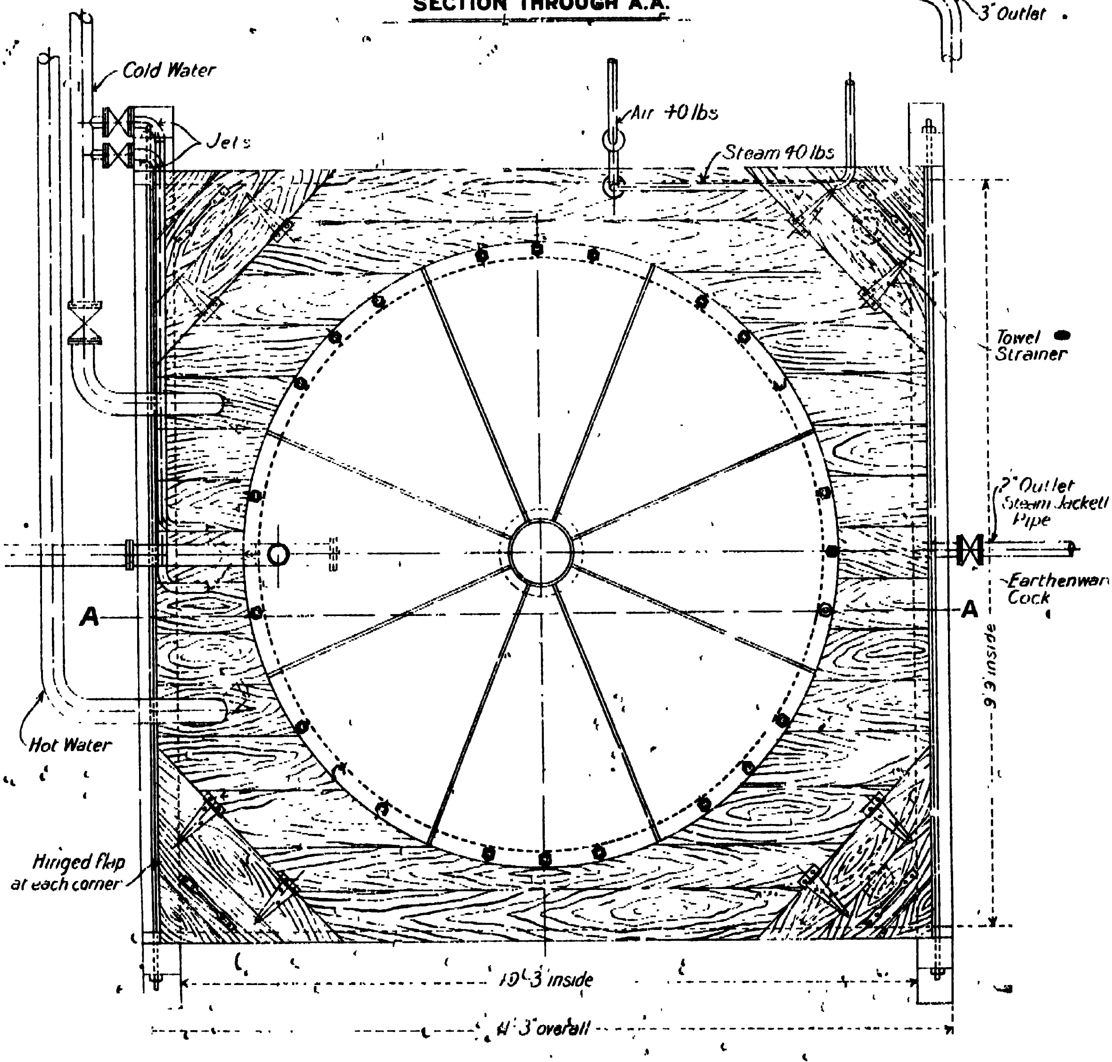
Flow of O.U.A. not to be started till stirrer is running. Charge to be heated to 70° C., maintained at 70° C. for 1 hour, then cooled to 25° C.

Time Period.		Time.		Temp. observed	Remarks.
Hr.	Min.	Hr.	Min.		
0	0				
	15				
	30				
	45				
1	0				
	15				
	30				
	45				
	0				
	15				
	30				
	45				
	0				
	15				
	30				
	45				
	0				
	15				
	30				
					S.G. of Product.

Signature..



SECTION THROUGH "A.A."



D.N.T. PELLETTING TUB & WASHER.

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a fall of 3 inches to the outlets. The vat is built of 3-inch tongued and grooved pitch pine, bolted together, all heads being recessed and wood plugs driven in. Across the two corners on the lower end are fixed two towelling filter cloths, which allow the wash-water to run through but which retain the pellite. A 3-inch regulus metal plug and seating controls the water outlet. The water is conveyed to the launder by means of 3-inch lead pipes. The necessary heating and agitation are effected by means of a 2-inch perforated spiral lead coil to which both air and steam, each at 40 lb. per sq. inch pressure, are connected. The pelleting is effected by means of two flat jets of water about 3/16 in. thick, one arranged 3 inches below the other, which impinge upon the descending vertical stream of DNT. The pelleter is supplied with cold and hot water.

In order to remove the fumes, the vat is provided at each corner with a cover of 1½-inch tongued and grooved wood, in the centre of which is a conical iron hood from which a vent pipe discharges the fumes direct to the atmosphere. All four corners of the vat are hinged, and may be lifted when necessary. The outlet to the next vat consists of a 2-inch earthenware cock with a steam jacketed 2-inch pipe.

Pelleting process.—The molten DNT is run by gravity from the nitration sight-box to the pelleting vat through a 3-inch steam-jacketed pipe, and is pelleted into 6 inches of cold water. The stream of DNT is met by the two jets of cold water, which causes it to divide into numerous small particles or pellets. After all the DNT has been pelleted, the acid water is allowed to drain off through the towel filters by means of a 3-inch lead pipe to an effluent launder constructed of acid resisting brick. This water, and the wash waters referred to hereafter, pass down the launder, through a labyrinth to allow any DNT to settle out, and finally to an effluent pit, where they, together with the wash-waters from the MNT plant, are neutralised with lime.

After receiving a second wash with a fresh charge of cold water the pelleted DNT is drained, and then melted by means of hot water from the storage tank and the steam coil, after which the melted DNT is run into the first of the three circular lead-lined washing vats situated just below, on the second of the four platforms of the washing plant.

Lead wash vats.—The 3 wash vats consist of circular vessels of 12 lb. lead, supported in skeleton frames of 2-inch pine. Each vat is 8 ft. 6 in. diameter × 2 ft. 9 in. high, and is provided with a conical steel fume hood made in sections which are bolted on to lead-covered angle-iron frames, a section of which may easily be removed, and one section of which is provided with a rectangular sight glass. A vent pipe made of 8-inch steel piping is attached to each hood, through which all fumes pass direct to the atmosphere. The temperature is maintained by means of a 1½-inch lead steam coil placed just off the floor of the vat, and a coil of perforated 1½-inch lead pipe is also provided for the purpose of air agitation. The two coils run in the form of a flat spiral together to the middle of the vat, where the steam coil passes through the bottom to a steam trap, whilst the air pipe is sealed off. Each of these pipes is supported about ¾ inch from the bottom of the vat to allow free passage for the liquid.

Cold water is supplied by a 2-inch line from the town supply, and warm water from the thermal storage tank on the MNT plant (see page 40).

Washing process.—The wash waters are syphoned off by two 2-inch lead pipes which are set at a predetermined height from the bottom of the vat, this height depending on the bulk of the DNT charge. The syphon is started by a full supply of cold water being turned through the long arm of the syphon. To prevent DNT being drawn up with the water, a horizontal flat lead plate is fixed 1 inch below the inlet of the syphon.

When the crude liquid DNT charge has been run into the first wash vat it has lost during pelleting, cold washing, and melting in hot water, between 80 and 90 per cent. of its acidity. The charge is next heated up with water from the hot-water storage tank, and well agitated for five minutes with air, after which it is allowed to settle for a few minutes, the wash water syphoned off and the DNT run into fresh hot water in the second wash vat, where the process is repeated. If the DNT should still be slightly acid after the second wash, as shown by examination of the wash water, the water is syphoned off and another wash is given in the same manner, fresh water being obtained from the storage tank.

A sample of DNT is then sent to the laboratory where it is tested for acidity, which should not at this stage exceed 0.02 per cent. calculated as H_2SO_4.

The washed DNT is next run into the third washer, which is used partly as a storage tank and partly as a preliminary drier, where it awaits removal to the driers.

Filtration and drying.—The washed and partially dried DNT is next pumped by means of a 2-inch centrifugal pump (driven by a 5 H.P. motor) through a 2-inch steam-jacketed line to the filter, from which it gravitates to two cast iron tanks, 7 ft. diameter × 2 ft. 6 in. deep, fitted with one flat coil and one helical coil for heating the charge.

The filter consists of a mild steel steam-heated vessel, 4 ft. 6 in. deep × 4 ft. 6 in. diameter. The filtration is effected by means of a perforated cast iron plate covered with felt. From the filter the DNT is run to the driers, which are maintained at a temperature of about 80° C. A sample is taken and tested in the laboratory for volatile matter, which must be below 0.25 per cent. The DNT is then run through a 3-inch steam-jacketed cock into the crystallisers.

Crystallisation.—The crystallisers (Fig. 62) consist of two pitch pine vats, 6 ft. 3 in. diameter × 2 ft. 9 in. deep × 3 in. thick. Each is provided with stirring gear consisting of a 3¼-inch diameter steel shaft, on which are keyed couplings to which are bolted three cast iron arms. These arms may be fitted at any angle and raised or lowered as required.

The stirring gear is driven by a 10 H.P. motor running at 955 r.p.m. The motor pulley is 11 inches in diameter, and drives a 40-inch diameter pulley fixed to a horizontal countershaft. To the latter are attached two 12-inch pulleys which drive the crystallisers by means of two 48-inch fast and loose pulleys operating bevel wheels with 14 and 51 teeth respectively; the speed of the stirrer is 17 r.p.m. The normal charge for each crystal-

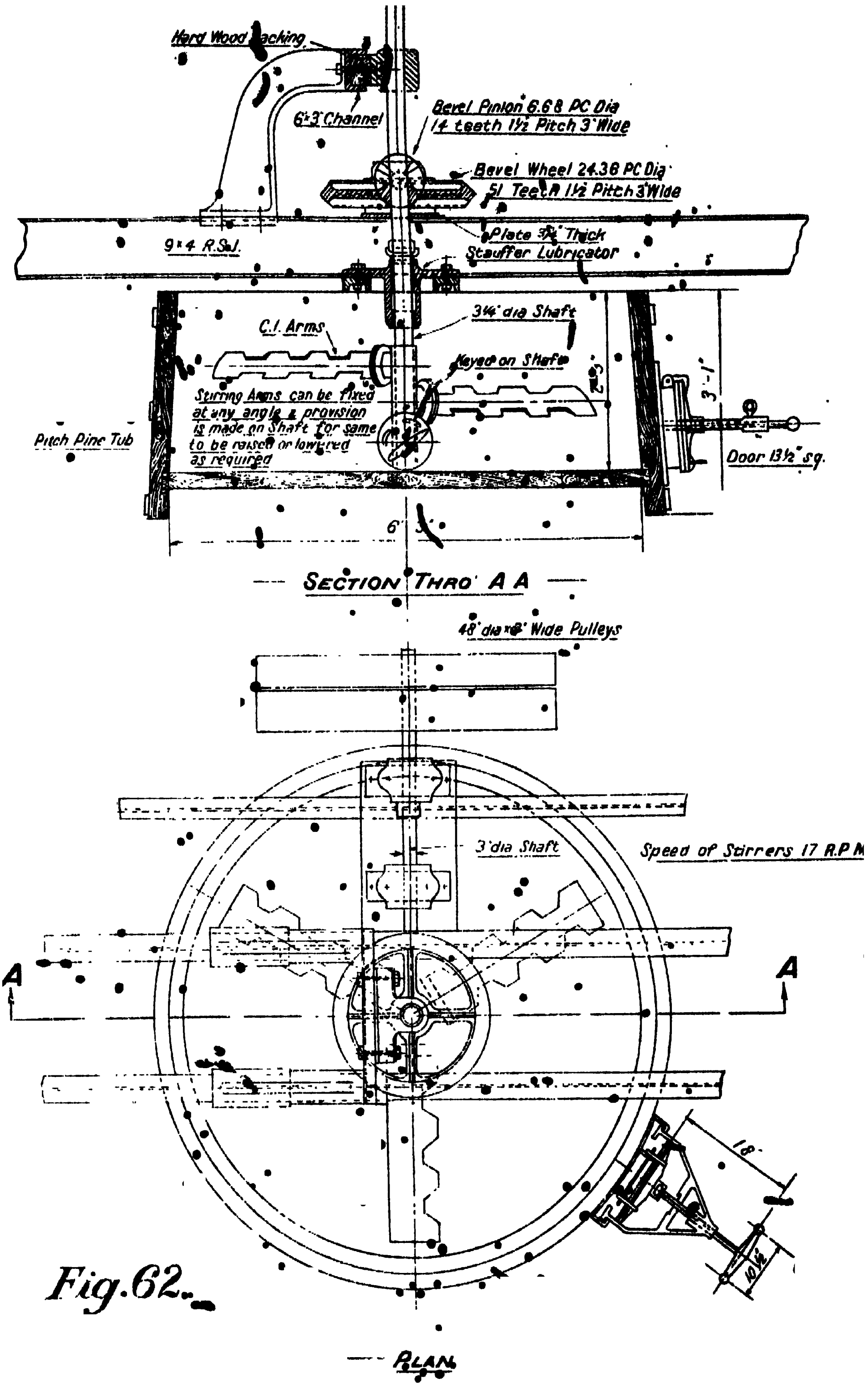
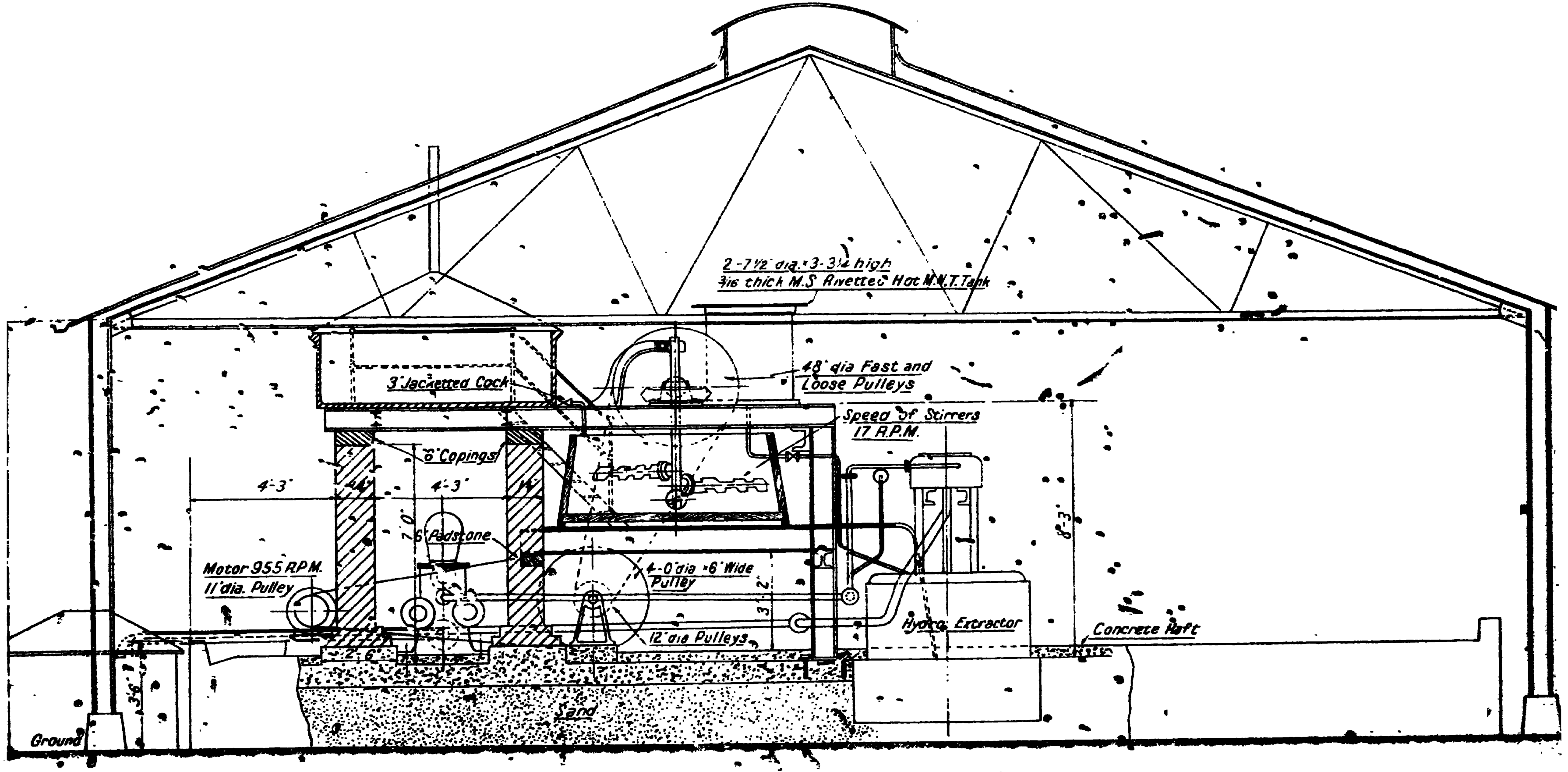


Fig. 62.

DETAIL OF CRYSTALLISER


2-7½" dia. × 3-3¼ high
3/16 thick M.S Rivetted Hot M.N.T. Tank
3' Jacketted Cock
48" dia Fast and Loose Pulleys
Speed of Stirrers 17 R.P.M.
6" Copings
4'-3"
4'-3"
7'-0"
6" Padstone
8'-3"
Motor 955 R.P.M. 11" dia. Pulley
4'-0" dia × 6" Wide Pulley
3'-2"
12" dia Pulleys
Hydro: Extractor
Concrete Raft
2'-6"
3'-6"
Sand
Ground

— CROSS SECTION THROUGH A A —

D.N.T. CRYSTALLISING PLANT

Fig. 63.

liser is about 1½ tons of crude DNT, which is run in at a temperature of approximately 55° C. The charge is allowed to crystallise with continued stirring for about 36 hours, the period varying according to the season of the year. When the temperature of the charge has fallen to 30°-35° C., and it is all in the form of fine dry crystals, the contents are discharged through a cast iron door, 13½ inches square, into a wooden launder, 9 in. wide × 8 in. deep, which feeds the centrifuges.

Centrifuging.—The centrifuging is effected by means of two 48-inch water driven hydro-extractors. The water is pumped from a mild steel tank, 6 ft. diameter × 5 ft. high, by a steam pump; the water pressure required is 200 lb. per square inch. The charge of crude DNT for each centrifuge is 1,000 lb., and this is whizzed for 30 minutes, during which time nearly all the oils of low melting-point are removed, leaving almost pure 2 : 4-dinitrotoluene. These oils, which consist of approximately 49 per cent. of 2 : 4-DNT, 37 per cent. of 2 : 6-DNT, 7 per cent. of other isomers, and 7 per cent. of MNT, flow away from the extractors through a gutter to a mild steel tank. The latter is fitted with a steam coil so that the oils may be kept in a fluid state whilst being pumped away to the storage tanks. Hot MNT is pumped along the line after each batch of DNT oils has been pumped away, in order to prevent any solidification taking place in the pipe line. The oils, which amount to about 46 per cent. of the crude DNT charged to the centrifuge, are delivered through a 2-inch line to the MNT storage tanks, where they mix with MNT to be used for TNT manufacture.

In order to prevent the copper gauze lining and the outer case of the centrifuge from clogging with solidified DNT, it is washed down with warm MNT before the introduction of a fresh batch, the centrifuge being allowed to rotate during this operation. The MNT for this purpose is stored in a mild-steel tank situated on the girders which support the crystalliser gearing.

The general arrangement of the drying and crystallisation plant is shown in Fig. 63.

Specification.—The 2 : 4-DNT left in the centrifuge consists of a pale yellow finely crystalline substance, which should conform with the following specification :—

1. The material is to be in the form of a light yellow crystalline powder free from mechanical impurities.

2. It is to be of such fineness that at least 95 per cent. will pass through a sieve of 20 meshes per lineal inch.

3. When heated to 100° C. it should melt to a transparent yellow liquid.

4. The crystallising point is not to be below 65.5° C.

5. The volatile matter as determined by the loss in weight at 60° C. is not to exceed 0.25 per cent.

6. The residue on ignition is not to exceed 0.05 per cent.

7. The material insoluble in benzene is not to exceed 0.10 per cent.

8. The acidity, calculated as sulphuric acid, is not to exceed 0.005 per cent.

Packing.—The 2 : 4-DNT is weighed direct from the centrifuges into iners which are packed in wooden boxes similar to those used for TNT. Each box holds 40 lb. of DNT, 50 boxes making one Lot, *i.e.*, one short ton.

Average laboratory figures for finished product:—

Dry setting point	67.7° C.
Volatile matter	0.022 per cent.
Insoluble in benzene	0.037 per cent.
Acidity	0.003 per cent.
Sulphated ash	0.013 per cent.

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